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PREFACE

This report presents the results of one phase of work performed by the Applied Mechanics Division of the Jet Propulsion Laboratory.

ABSTRACT

Graphite epoxy material for a nickel cadmium battery cell case had previously been evaluated and determined to perform in the simulated environment of the battery. Basic manufacturing method refinements were performed to demonstrate production feasibility. The various facets of production scale-up, i.e., process and tooling development, together with material and process control, have been integrated into a comprehensive manufacturing process that assures production reproducibility and product uniformity. Test results substantiate that a battery cell case produced from graphite epoxy pre-impregnated material, utilizing the internal pressure bag fabrication method, is feasible

In addition to improvements in manufacturing processes, the case testing program underwent enhancement in case confinement, environment application and recording of observations.

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SECTION I

INTRODUCTION

A program was conducted to identify and evaluate approaches which could affect significant weight savings when applied to NiCd cells and batteries (Ref. 1-1).

A graphite epoxy (G/E) cell case has been developed for a nickel cadmium battery (Ref. 1-2) and has been evaluated in the performance environment of the battery. Preliminary evaluation of the G/E cell yields a case resistant to the potassium hydroxide (KOH) electrolyte, resistant to gaseous diffusion through the case material, capable of withstanding internal pressures in excess of 300 psig, and weighing an order of magnitude less than the current stainless steel variety (see Fig. 1-1).

The existing units are 20 A-hr, NiCd cells. These cells have a power density of 12 to 15 Wh/lb. The electrodes operate in a KOH electrolyte, generate oxygen during the charge cycle and can generate an internal pressure on the order of 70 psig.

The configuration of the existing case, shown in Fig. 1-2, is a rectangular "cigarette pack" consisting of 0.025-in.-thick annealed Type 304L stainless steel. The cell, due to its 70 psig internal pressure and hazardous fluid, must be man-rated as a pressure vessel. Man-rated margins of safety of 2.25 on yield and 4.0 on burst establish design requirements for either construction material.

This program developed manufacturing and processing techniques in the fabrication and evaluation of 160 G/E cases. Programmed Composites Company, Inc. was selected by the Jet Propulsion Laboratory to manufacture the cases as a result of a competitive solicitation.

Early attempts to fabricate the cases resulted in an irregular product, premature failures upon burst testing, and an unacceptably high part-rejection rate. Manufacturing process improvements and corrected testing conditions corrected these deficiencies and minimized production time and product rejections.

The processing improvements included closer definition of raw material requirements, and tooling modification to more uniformly apply pressure and processing improvements.

Case testing and evaluation of process control specimens (tag ends) indicated that parts, manufactured to the described process, (Ref. 1-3) yielded reproducible hardware capable of withstanding design loads in the anticipated environment (see Table 2-1). Experimental verification of case Burst pressures has been conducted and representative cases are being subjected to alkali exposure and cyclic testing.

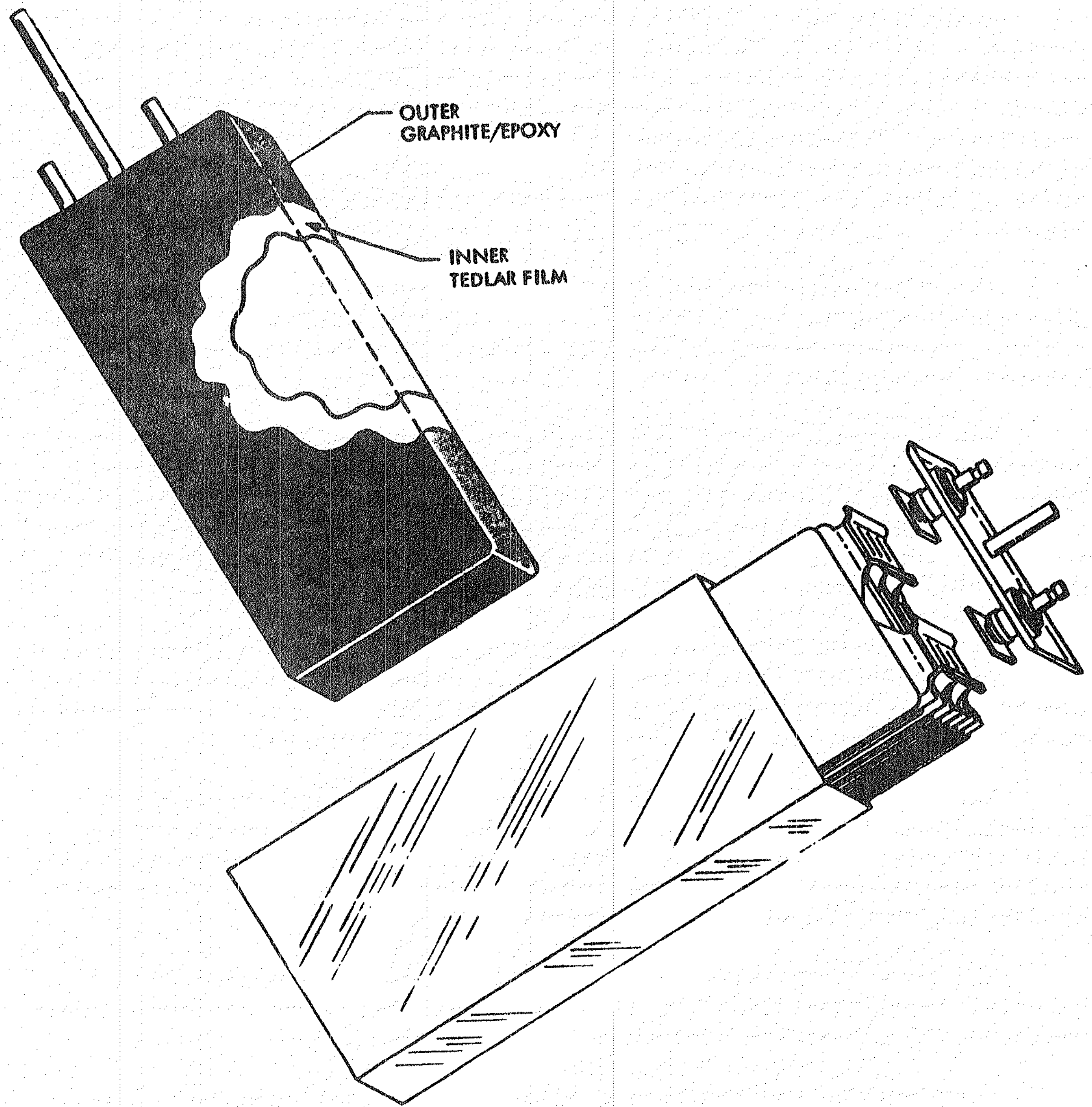

Figure 1-1. G/E Cell Case and Current Stainless Steel Case

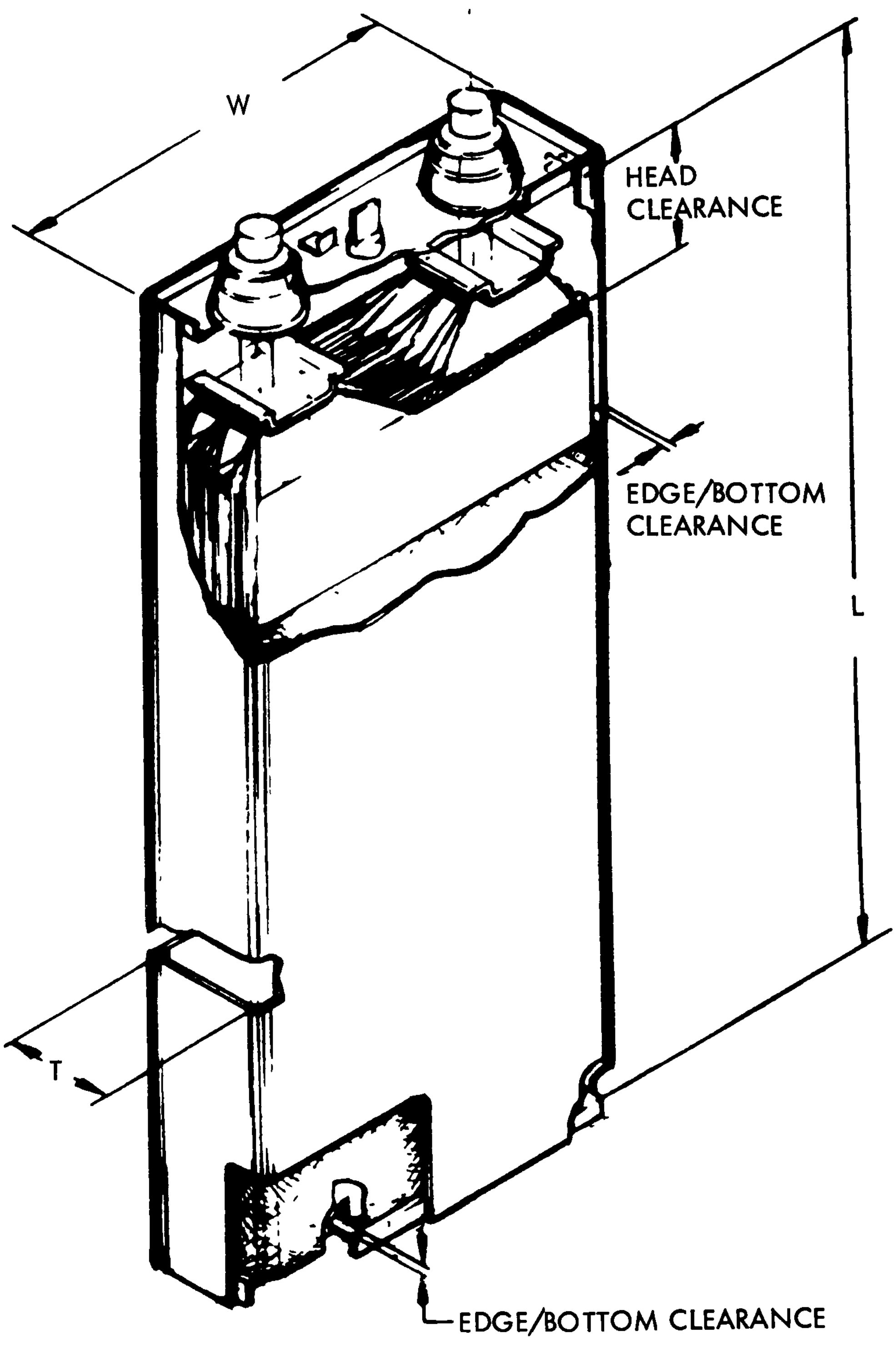

Figure 1-2. Standard Cell

SECTION II

MATERIALS

Several materials were considered as possible substitutes for stainless steel to reduce case weight. They included titanium, Zircalloy II, 4130 hot-rolled steel foil, Inconel 718 and G/E composite (Thornel 300). Graphite epoxy, with a significantly lower density and equivalent strength, was selected as the cell case material. Because of the cell burst requirement, a high-strength fiber was selected. A thin prepreg was desirable because it permitted sufficient ply build-up yet minimized wall thickness and weight. The Thornel 300 fiber, produced by Union Carbide, was selected because it offered the best combination of properties and product uniformity in thin tape (0.0025 in.). Ferro Corporation, because of its ability to reliably provide a uniform 2.5 mil prepreg with a high-performance resin (Ref. 2-1), was selected to perform the impregnation. (Design requirements for test specimens and case strength were determined using mechanical properties obtained from the Advanced Composites Design Guide, Ref. 2-2.) The resin system selected was Ferro's CE 9015, a 350°F cure low outgassing, epoxy system.

The cover was molded from a commercially available chopped G/E molding compound, EM-7125, which was supplied by U.S. Polymeric.

To eliminate any porosity of liquids or gases through the G/E case wall, a 4-mil layer/liner of Tedlar, (polyvinyl fluoride) film was located on the internal surface of the case (shown in Fig. 1-1).

The film, as supplied by Du Pont, possesses bondable or adherable surfaces, i.e., capable of being adhesively bonded to a different surface. Some subsequent processing had removed or destroyed this bondable surface. The surface has been regenerated by the use of Tetra Etch, a caustic etchant for preparing Teflon surfaces for bonding. Tetra Etch is a produce of W. L. Gore & Associates. Tetra Etch was applied per manufacturer's directions and followed with a toluene and detergent wash. A distilled-water flush completed the surface preparation.

To determine the bondability of the Tedlar (which constitutes the inner wall of the case), standard lap shear tests were performed. These consisted of aluminum to Tedlar-covered G/E skin; G/E molding compound to Tedlar-covered G/E skin; and G/E molding compound to G/E skin (no Tedlar). The results in Table 2 show an improvement in bonding strength with the Tedlar, and compare favorably to previous work on the program (Ref. 1-1).

Samples of 4-mil-thick Tedlar plastic were submitted to PJB Laboratories, Pasadena, for permeability determination. The samples were conditioned at 50% relative humidity prior to testing. The permeability determination was made using ASTM D1434-75, Standard Methods of Test for Gas Transmission Rate of Plastic Film and Sheeting, Method M (Ref. 2-3).

Table 2-1. Manufacturing Data of Graphite Epoxy Cases

Serial number	Case weight (g)	Specific gravity	Fiber volume	Void content
001	30.95	1.55	60.24	1.3
002	32.88	1.57	63.2	2.1
003	30.10	1.60		
004	31.38	1.57	59.1	2.0
005	34.01	1.54	54.3	1.1
006	29.84	1.56	57.3	1.9
007	32.16	1.51		
008	30.54	1.57		
009	31.76	1.59		
010	32.11	1.57		
011[a]	32.58	1.58	57.7	0.7
012[a]	31.91	1.57		
013[a]	31.69	1.60		
014[a]	31.78	1.56	57.3	1.1
015[a]	32.44	1.56	57.9	0.4
016	32.44	1.56		
017	32.00	1.56		
018	30.65	1.57		
019	30.67	1.57	58.5	0.6
020	33.15	1.58	59.7	0.4
021	31.20	1.57		
022	31.53	1.57		
023	31.25	1.56	56.2	0.8
024	30.75	1.58	61.03	0.7
025	31.48	1.57		
026	32.40	1.55		
027	32.70	1.53		

[a]Vacuum degassed before cure.

Table 2-1. (contd)

Serial number	Case weight (g)	Specific gravity	Fiber volume	Void content
028	32.49	1.55		
029	32.47	1.54		
030	32.38	1.51		
031[b]	32.42	1.54		
032[b]	31.63	1.55		
033[b]	31.47	1.58		
034[b]	31.06	1.54		
035[b]	32.05	1.58		
036[c]	29.73	1.56		
037[c]	30.16	1.58		
038[c]	30.37	1.57		
039[c]	37.83	1.58		
040[c]	34.37	1.56		
041	29.85	1.55		
042	30.93	1.58		
043	31.19	1.56		
044[b]	32.52	1.56		
045[b]	33.13	1.56		
046[b]	31.51	1.55		
047[b]	31.32	1.55		
048	30.31	1.55		
049	31.42	1.56		
050	31.07	1.57		
051	30.09	1.57		
052	31.18	1.55		
053	31.86	1.56		
054	31.69	1.58		

[b] Inside narrow side coated with 3M 2216 epoxy adhesive.

[c] Interior surfaces coated with RTV 632 silicon rubber.

Table 2-1. (contd)

Serial number	Case weight (g)	Specific gravity	Fiber volume	Void content
055	33.18	1.57		
056	32.17	1.57		
057	32.08	1.58		
058	31.16	1.55		
059[b]	31.91	1.59		
060[b]	32.32	1.58		
061[b]	32.08	1.56		
062[b]	30.69	1.58		
063[b,c]	31.49	1.59		
064[b]	31.73	1.59		
065[b,c]	31.94	1.56		
066[d]	30.88	1.55		
067[d]	31.84	1.54		
068[d]	31.53	1.55		
069[d]	30.99	1.59		
070[d]	32.13	1.56		
071[d]	30.90	1.59		
072[d]	30.29	1.57		
073[d]	32.11	1.57		
074[d]	32.14	1.55		
075[d]	31.88	1.56		
076[e]	39.15	1.56		
077	38.42	1.58		
078	36.25	1.57		
079	36.75	1.57		

[b] Inside narrow side coated with 3M 2216 epoxy adhesive.

[c] Interior surfaces coated with RTV 632 silicone rubber.

[d] 0.001 in.-thick Tedlar film secondarily bonded to interior of narrow side using 3M-2216 epoxy adhesive.

[e] 10-ply case.

Table 2-1. (contd)

Serial number	Case weight (g)	Specific gravity	Fiber volume	Void content
080	39.50	1.58		
081	35.69	1.58		
082	36.21	1.58		
083	37.04	1.58		
084	35.38	1.59		
085	35.99	1.59		
086	36.47	1.60		
087	36.81	1.57		
088	35.48	1.59		
089	35.53	1.57		
090	36.60	1.54		
091	34.90	1.57		
092	37.36	1.57		
093	37.12	1.57		
094	37.29	1.56		
095	34.90	1.54		
096	37.00	1.57		
097	35.45	1.59		
098	37.40	1.56		
099	35.80	1.58		
100	35.71	1.58		
101	36.58	1.58		
102	38.05	1.55		
103	36.85	1.57		
104	38.84	1.55		
105	36.51	1.58		
106	37.30	1.57		
107	37.19	1.59		

Table 2-1. (contd)

Serial number	Case weight (g)	Specific gravity	Fiber volume	Void content
108	36.34	1.57		
109	37.73	1.56		
110	36.89	1.55	56.36	1.1
111	35.21	1.57	58.7	0.7
112	34.99	1.59	61.98	0.5
113	36.10	1.55		
114	38.57	1.59		
115	35.20	1.54		
116	35.26	1.55	56.1	1.2
117	35.58	1.57		
118	36.07	1.56		
119	35.53	1.56	59.4	1.4
120	35.50	1.56		
121	35.02	1.56		
122	36.47	1.56		
123	37.54	1.56		
124	37.33	1.52		
125	37.72	1.55		
126	37.70	1.56		
127[f]	39.13	1.57/1.53[g]	60.2	1.0
128	41.11	1.55/1.50	59.3	0.8
129	38.34	1.56/1.56	58.0	1.5
130	40.49	1.56/1.53	58.9	0.7
131	38.24	1.56/1.55	59.3	0.8
132	40.57	1.58/1.50	49.9	1.2
133	41.69	1.56/1.53	47.0	0

[f] Co-cured 4-mil thick Tedlar film on interior surfaces.

[g] First specific gravity represents narrow side; second represents wide side.

Table 2-1. (contd)

Serial number	Case weight (g)	Specific gravity	Fiber volume	Void content
134	40.18	1.57/1.53[g]		
135	39.66	1.59/1.55		
136	39.61	1.55/1.53		
137	36.49	1.53/1.55		
138	38.17	1.55/1.55		
139	39.81	1.55/1.51		
140	39.27	1.55/1.52		
141	39.57	1.57/1.50		
142	41.05	1.57/1.50	58.5	0.6
143	39.11	1.54/1.51		
144	37.58	1.57/1.57		
145	36.88	1.57/1.58		
146	39.99	1.57/1.52		
147	40.54	1.57/1.53		
148	41.06	1.57/1.51		
149	39.68	1.53/1.53		
150	39.87	1.56/1.52	58.1	0.6
151	40.09	1.56/1.52		
152	41.61	1.56/1.50		
153	38.48	1.58/1.55		
154	38.53	1.54/1.55		
155	42.43	1.54/1.50		
156	42.37	1.58/1.53		
157	38.55	1.46/1.53		
158	39.30	1.53/1.53		
159	39.21	1.55/1.50	46.1	1.4
160	42.10	1.57/1.51	58.5	1.1

[g]First specific gravity represents narrow side; second represents wide side.

Table 2-2. Lap Shear Strength of Tedlar-Covered G/E

Sample	Lap shear strength (psi)
Aluminum to Tedlar-covered G/E skin	2170
	1882
	2052
	2144
	1860
	2020
G/E Molding compound to Tedlar-covered G/E skin	2319
	1921
	2004
	1725
	1850
	1850
G/E Molding compound to G/E skin	1899
	1588
	1450
	1646

Ten separate determinations of the permeability were made and the average value was 1.9×10^{-7} cc/atm-sec, with a standard deviation of 1.1%. The dimensions of the test pieces were 4 mil thick with a test area of 9.6 cm^2.

SECTION III

FABRICATION

Hardware was fabricated in female tooling with internal pressure equal to or greater than anticipated service pressure. This fabrication technique yielded dense, high-quality cases with a minimum of molded-in thermal stresses due to more evenly distributed individual fiber hoop stresses. Therefore, the tooling concept involves a matched metal female die with an internal high-pressure elastomeric bladder (see Fig. 3-1).

The mold was used for fabricating the expandable bladder. General Electric RTV-630 silicone rubber was used. An aluminum dummy plug was introduced, and located and bolted in place to establish an approximate 1/8-in. bladder wall thickness. An additional post-curing of four to five hours in the free state in an air circulating oven at 350°F completed the shrinking to approximately 0.080 in. smaller than the external metal tool.

The compression die for molding the end caps (Fig. 3-2) was designed with the thickness, or the "Z" axis dimension, as the variable. As-molded parts made in this manner were applied to a trim fixture and machined on their back face to an absolute dimension, weight and parallelism. This trim fixture was also used to locate the drilled holes for electrode terminals and the vent/fill tube. The last operation in the trim fixture established the end radius and width dimension allowing for a nominal glue line.

The approved manufacturing plan is contained in Appendix A.

A. PROCESSING

An aluminum bar was clamped vertically in a bench vise and the rubber bladder was then nested over, thus rigidizing the rubber in preparation for fabrication of the Tedlar liner by heat-sealing techniques and layup of the prepreg tape.

A longitudinal 0°, 19 in.-long wrap-ply was applied with finger pressure to the supported rubber bladder (and sealed Tedlar liner) near one of the flanges. This manipulation was continued until all of the wrinkles were removed and the ply was adhered on one broad face, across the bottom, and up the far side to the opposite flange. A 1-in.-wide strip, 21 in. in length, was applied in a similar manner down a side, across the bottom, and up the opposite side, thus completing the layup application of one longitudinal ply. (Note that the bottom receives a 90° crossed, second ply by this method.) Three, 3-in.-wide patterns, 8-1/2 in. long, were then longitudinally spliced together to form a 90° second ply. The extra one-half inch provided by this pattern was used as an overlap or tab splice at the ends of the hoop fibers. The location of the splice was noted and each hoop ply applied thereafter had its splice on an adjacent corner (see Fig. 3-3). These two methods

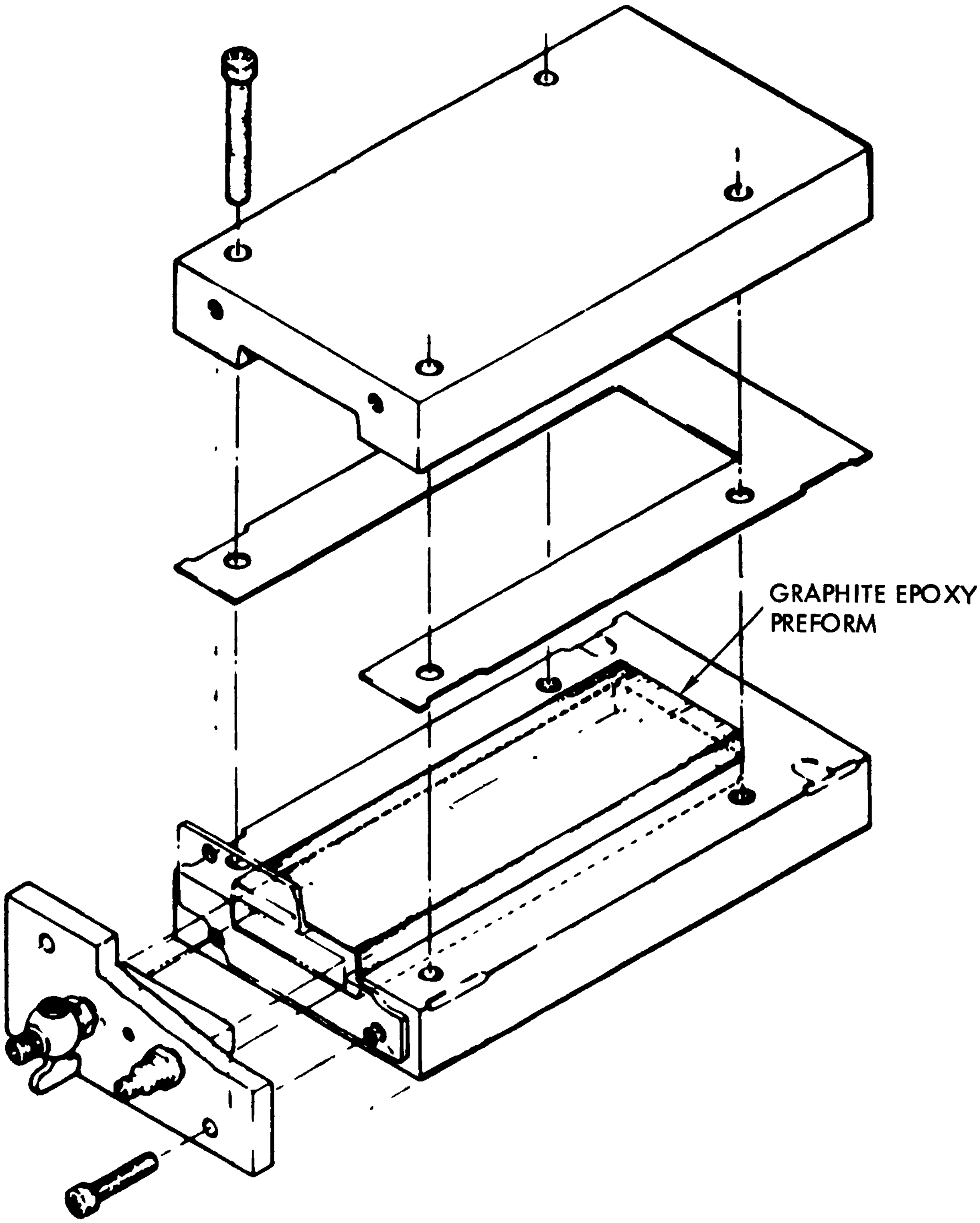

Figure 3-1. Cell Case Tooling

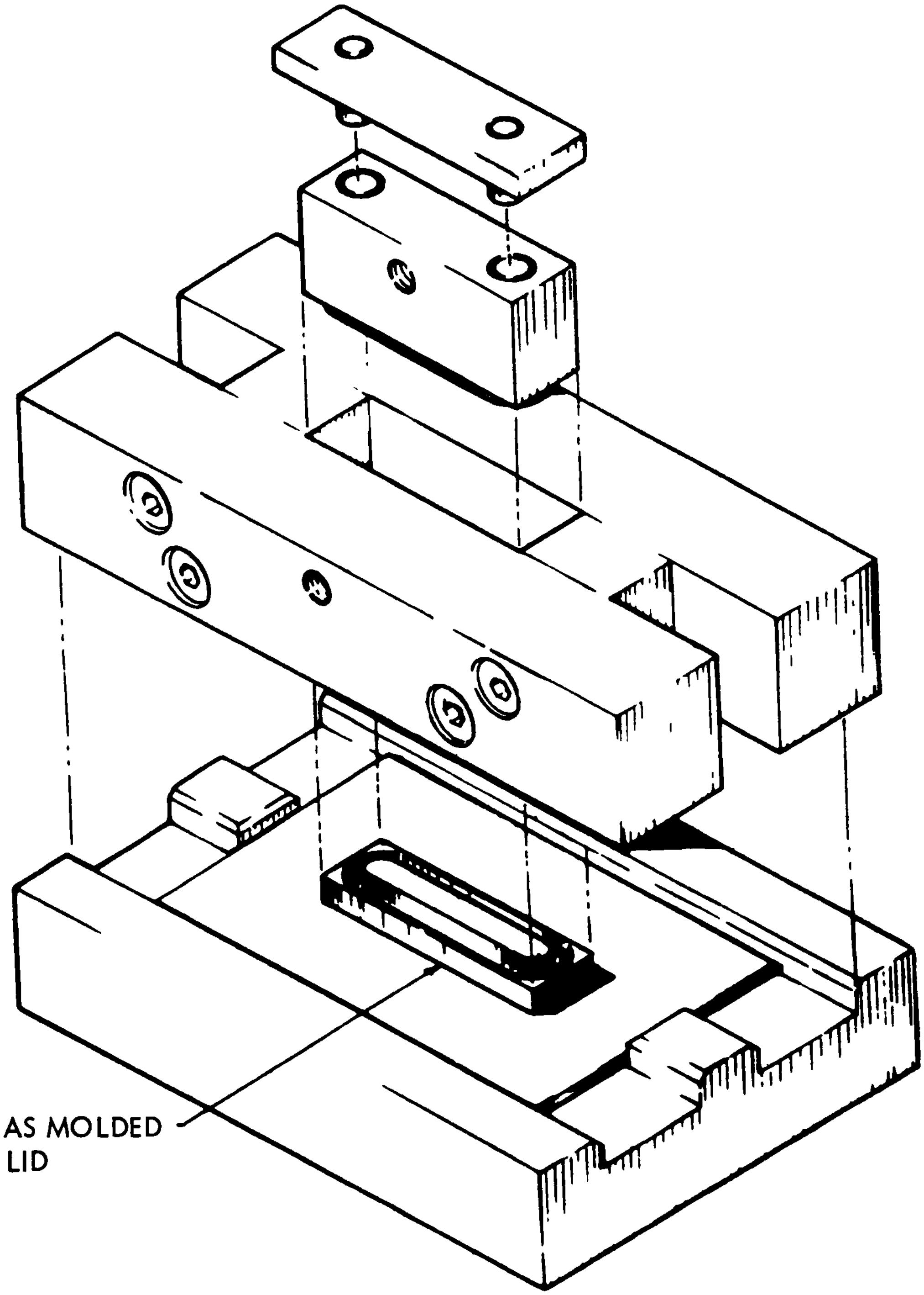

Figure 3-2. Cell Cover Tooling

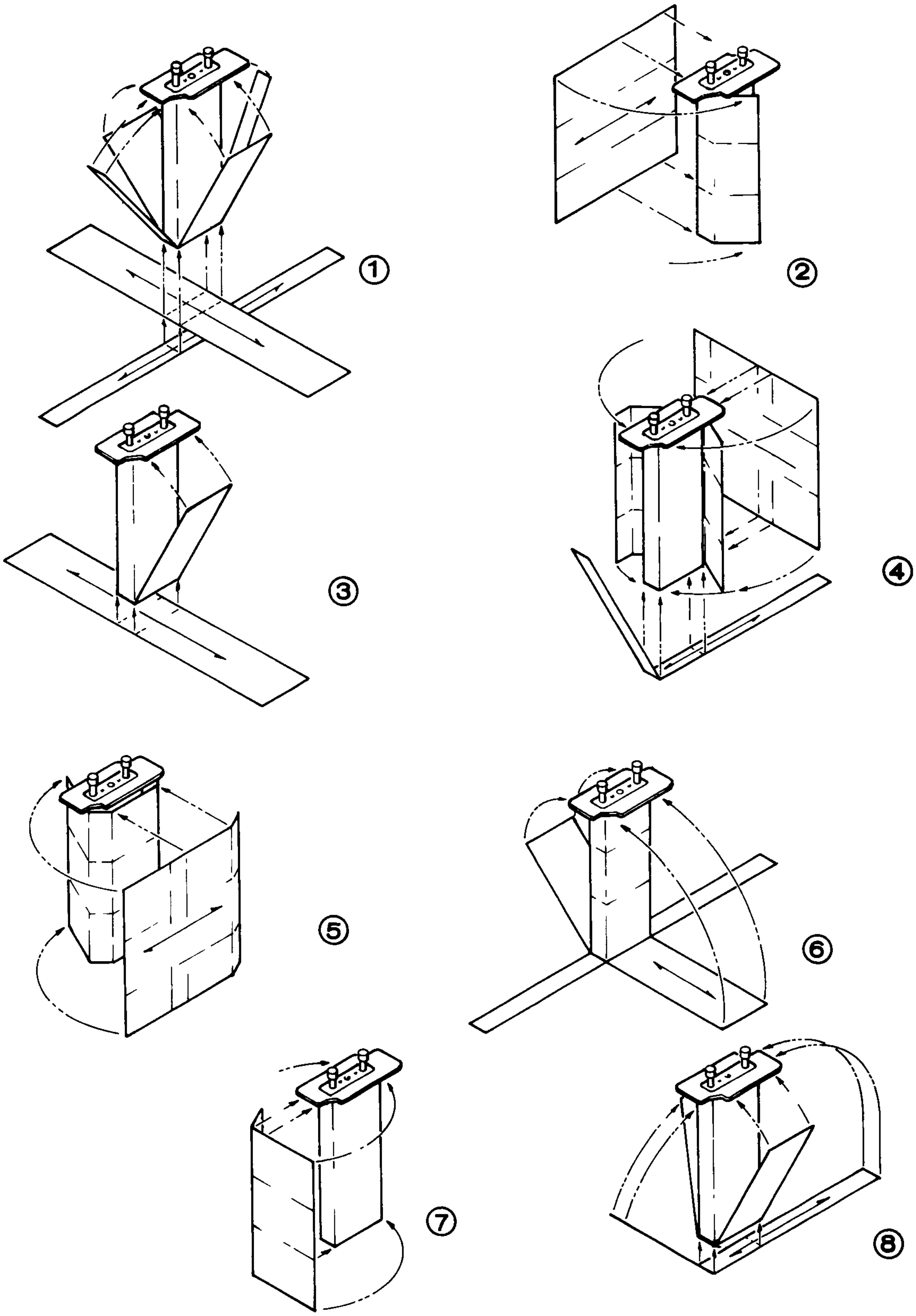

Figure 3-3. Layup Pattern of G/E Prepreg

were repeated alternately until the desired number of plys were incorporated.

The aluminum bar was removed and the preform/bladder assembly was positioned in the tool. The manifold end cap was located and bolted in position. The loaded mold was then placed in a heated platen press and the load was advanced to 3000 lbs. The air pressure, regulated to the required value, was charged to the inside of the bladder and set for the duration of the cure. The high pressure within the bladder caused it to expand uniformly against the G/E preform, which in turn, was compressed against the aluminum mold surface. Heat delivered by the press platens heated the preform, causing the resin to melt. Trapped air bubbles in the preform were expelled through a series of vents located along the mold split line. Curing was continued in this state for 60 to 90 minutes. Demolding was accomplished hot. The cannister was now trimmed to length but was scuff sanded on its bond surface. The Tedlar film surface was treated with Tetra Etch prior to bonding.

B. TRACEABILITY

Throughout the program, records were maintained so that fabrication time and yield rates could be established. The initial phase of the fabrication program showed higher labor time and excessive rejection rates. This was attributed to processing, the developmental nature of the program, and a lack of definitive requirements for the raw material characteristics for the G/E prepreg material.

Upon resolving the processing problems, which incorporated definitive raw material characteristics, the rejection rate was less than 5%. This reduction in rejection rate was naturally reflected in fabrication time. The actual fabrication time for case layup, cure, cover molding, and match machining of cover and case was approximately five hours per unit.

SECTION IV

PROCESSING IMPROVEMENTS

A. CASES

The fabrication procedure for the battery cell case was defined in the JPL manufacturing process specification (Ref. 1-3) and the raw material requirements in the JPL material specification (Ref. 2-1). As the case fabrication program proceeded, it became apparent that the tolerance requirements for the raw material characteristics were too liberal. Although the Ferro Corporation prepreg material, CE-9015, met the specification requirements, the normal variation in resin content was the primary cause of the processing problems. The high resin content material (48-50%) processed in accordance to specification resulted in resin puddles on the case interior walls. These puddles became rigid after cure (Fig. 4-1) and would cause damage to the silicone rubber bladder during attempts to remove the bladder. However, when low-resin content material (40-42%) was processed, the case showed evidence of being porous when subjected to a 10-psig pressure leak test.

The processing of high-resin content material and the associated problems were controlled by modifying the silicone bladder. A metal shim (2-1/2 in. x 10 in. x 0.005 in.) was incorporated into the flat wall of the bladder at the time of bladder fabrication (Fig. 4-2). This improvement in the silicone bladder allowed cases to be manufactured with uniform wall thickness without resin-rich puddles. Although visually acceptable cases were produced, the high-resin content did not eliminate the porosity problem and resulted in a yield rate that was too low.

The porosity manifested itself as a weeping of test fluid (water) through the case walls during attempts to burst test fabricated cases. This weeping was not a sustained flow of fluid that would indicate structural failure, but did prevent the cases from sustaining pressures in excess of 150-200 psi. Table 4-1 summarizes the burst test results of the first 76 cases. Cases 061 through 071 indicated a lack of ability to contain elevated pressures while not sustaining structural damage.

Various techniques were attempted to correct the porosity of the cell case wall which was exaggerated by low-resin content material. The first approach was a coating of 3M Company's 2216 epoxy resin to the internal surface of the walls. This did not appreciably reduce case porosity. Later, a 1-mil layer of Tedlar film was bonded to the 0.86 wall using the 3M-2216 epoxy resin. With the application of the coating and film, the battery cases routinely passed the pressure leak test.

The application of a 4-mil thick Tedlar liner to the internal surfaces of the battery case proved to be most acceptable. A technique for forming and installing the 4-mil thick Tedlar was developed. Utilizing the aluminum male plug to support the silicone rubber bladder, a precut pattern (shown in Fig. 4-3) of 4-mil thick Tedlar film was shaped and

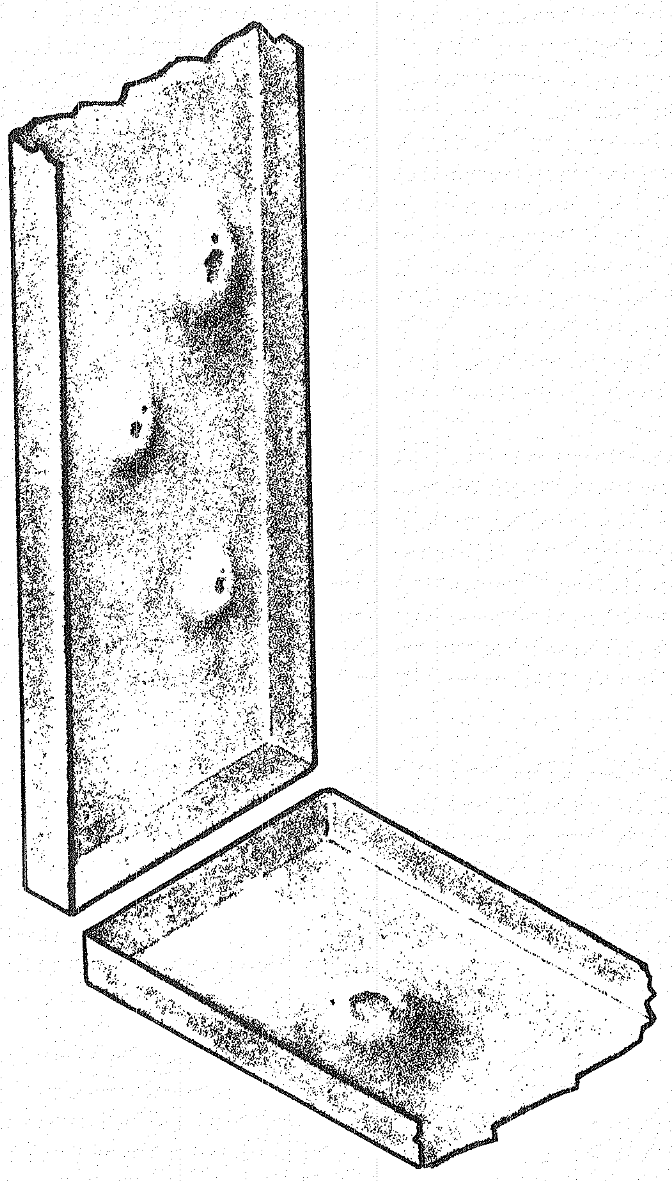

Figure 4-1. Cured Resin Puddles on Case Interior

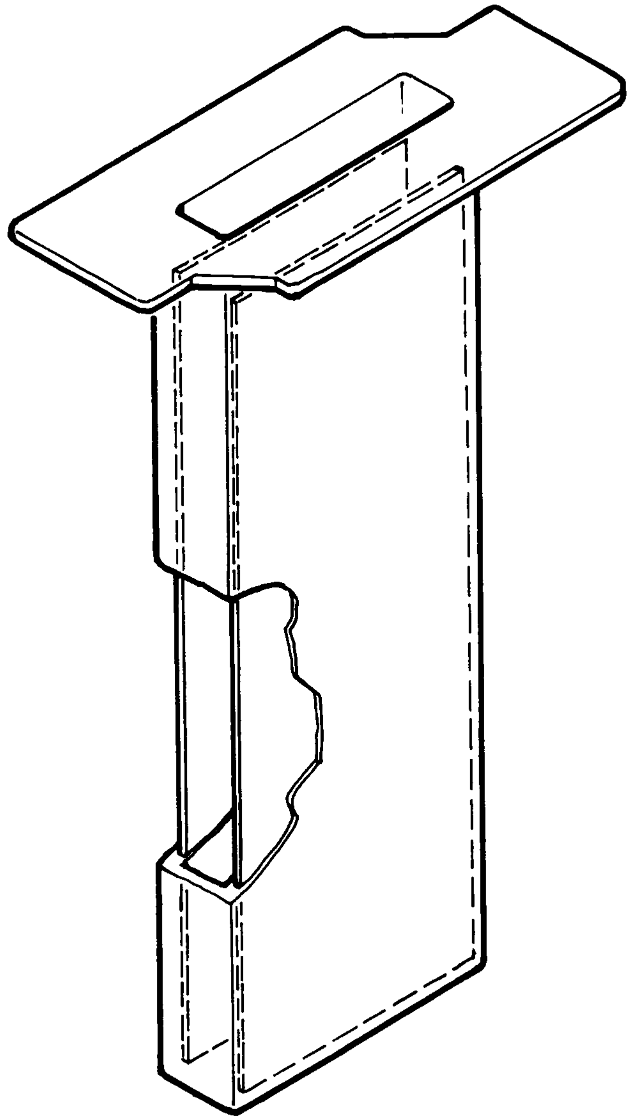

Figure 4-2. Metal Shim Reinforced Walls of Silicon Bladder

Table 4-1. Burst Test and Helium Permeability of the First 76 Cases

Serial number	Burst pressure (psi)	Helium permeability (scc/sec)	Remarks
001[a]	240	1.2×10^{-5}	Minute delamination (MD)
002	50	Gross	Inadequate test support (ITS)
003[a]	235	1.7×10^{-6}	MD
004	80	Gross	Gross delamination ITS + (GD)
005	140	Gross	MD
006	110	1×10^{-5}	ITS
007	220	No Leak	GD
008	255	1.2×10^{-5}	MD
009	225	Gross	Bottom Failure
010	210	Gross	GD
011[a]	260	No Leak	GD
012	170	Gross	GD
013	210	No Leak	--
014	210	Gross	--
015[a]	110	6×10^{-5}	GD
016	190	2.6×10^{-7}	--
017	180	2.2×10^{-6}	--
018	205	1.3×10^{-4}	--
019	180	3.8×10^{-4}	--
020	175	1.2×10^{-6}	--
031	--	Gross	--
032	170	2.1×10^{-8}	--
033	130	Gross	--
034	180	Gross	--
035	195	Gross	--

[a]Repaired and retested

Table 4-1 (Contd)

Serial number	Burst pressure (psi)	Helium permeability (scc/sec)	Remarks
061	150	--	Narrow-side structural failure
062	215	--	Porosity on narrow side
063	230	--	Porosity on narrow side
064	340	--	Delamination (case/cover)
065	170	--	Delamination (case/cover)
066[b]	250	--	Porosity, no failure
067[b]	330	--	No failure
068[b]	280	--	No failure
070[b]	280	--	Minute porosity, no failure
071[b]	350	--	Porosity, no failure
072[b]	430	--	Side burst
073	210	--	Side burst
074	370	--	Side burst
075	310	--	Side burst
076	310	--	Side burst

[b]By P.C.

heat tacked to hold its shape. The eight ply of G/E prepreg material was laid-up in contact over the Tedlar film. The combined layup was then co-cured using the same procedure that was used to cure the graphite epoxy. The resulting composite case was subjected to the various pressure and environmental tests and complied to these requirements. The yield of battery cases produced with a 4-mil thick Tedlar film utilizing the metal-shimmed silicone bladder has been 95%. A weight penalty of one gram was incurred by incorporating the Tedlar film into the case.

The raw material characteristics should be controlled to closer tolerance than the specification. For example, based upon the experience gained during the course of this program, it is recommended that resin content be held to 43-47% and volatile content to 3%, at the maximum. By imposing closer controls on the raw material and maintaining

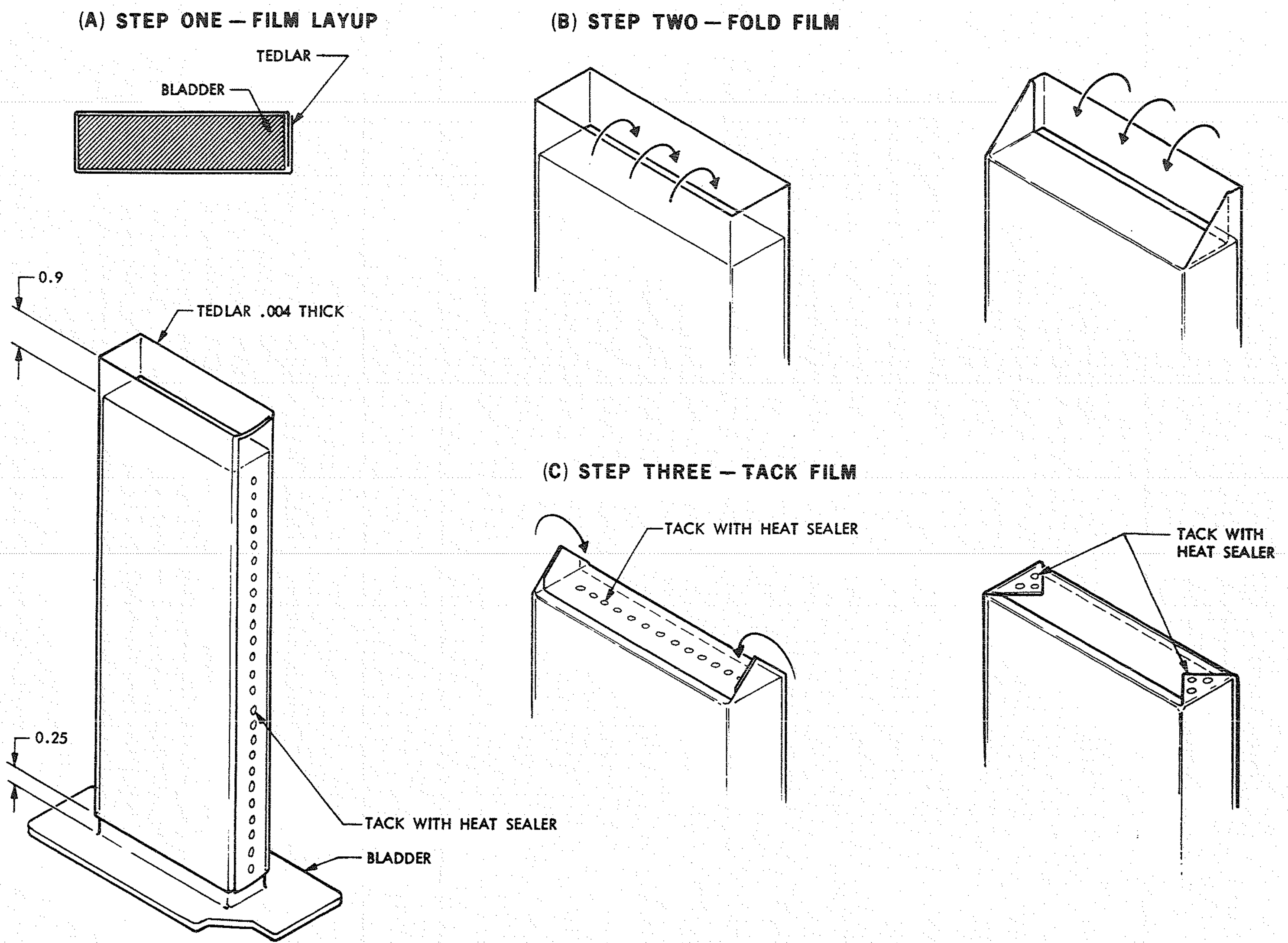

Figure 4-3. Layup Pattern of Tedlar Film

rigid process controls, a yield of battery cell cases could be maintained at about 95%.

B. COVERS

Since the covers must meet certain electrical requirements in addition to sealing the case, further design work was necessary. Each of the two terminals passing through the cover must be electrically insulated, as well as contain the anticipated operating pressure. This design was developed and is illustrated in Fig. 5-1. Appendix B illustrates the design in detail.

SECTION V

TESTING

During the burst-testing phase of the program, it was observed that case failure occurred in delamination of the case. This delamination occurred in the ply closest to the cover bond line (Fig. 5-2). Initial observations incorrectly reported these failures as adhesive failures. An examination of Table 4-1 shows that the principle failure method for the first 35 cases was actually ply delamination.

The joint design, test fixture, and test pressurizing procedures were reviewed. Recommendations were made to design and fabricate a fixture to completely contain the battery case in a mode similar to that in service (Fig. 5-3). Also, changes in the application of pressure (no surge of pressure) during burst testing were suggested. In addition, the configuration of the bonded cover created a stress riser. By providing a tapered cover and inserting it into the case in an inverted position with a generous adhesive fillet, stress at the bond area was reduced. Subsequent burst testing has failed to exhibit case delamination as a failure mechanism.

Initial-case burst testing had been conducted with an interior aluminum shim machined to fit the inside dimensions of each case. The shim was provided to support the case during the burst test (Ref. 1-2). These spacers were machined from aluminum stock at a rate of four hours of machining time per case. Since some of the test cases would require exposure to a KOH environment, an alternative material would have been required.

To reduce the cost of the spacers and achieve KOH resistance, a cast polyester shim was developed (Ref. 5-1). The polyester spacer was found to be KOH resistant and also reduced the cost of each spacer from $84 for the aluminum to $15 for the polyester. Polyester spacers were used in tests for cases 020 through 076. Subsequent refinements in the test jig design rendered the shims unnecessary and subsequent burst tests have been conducted without them.

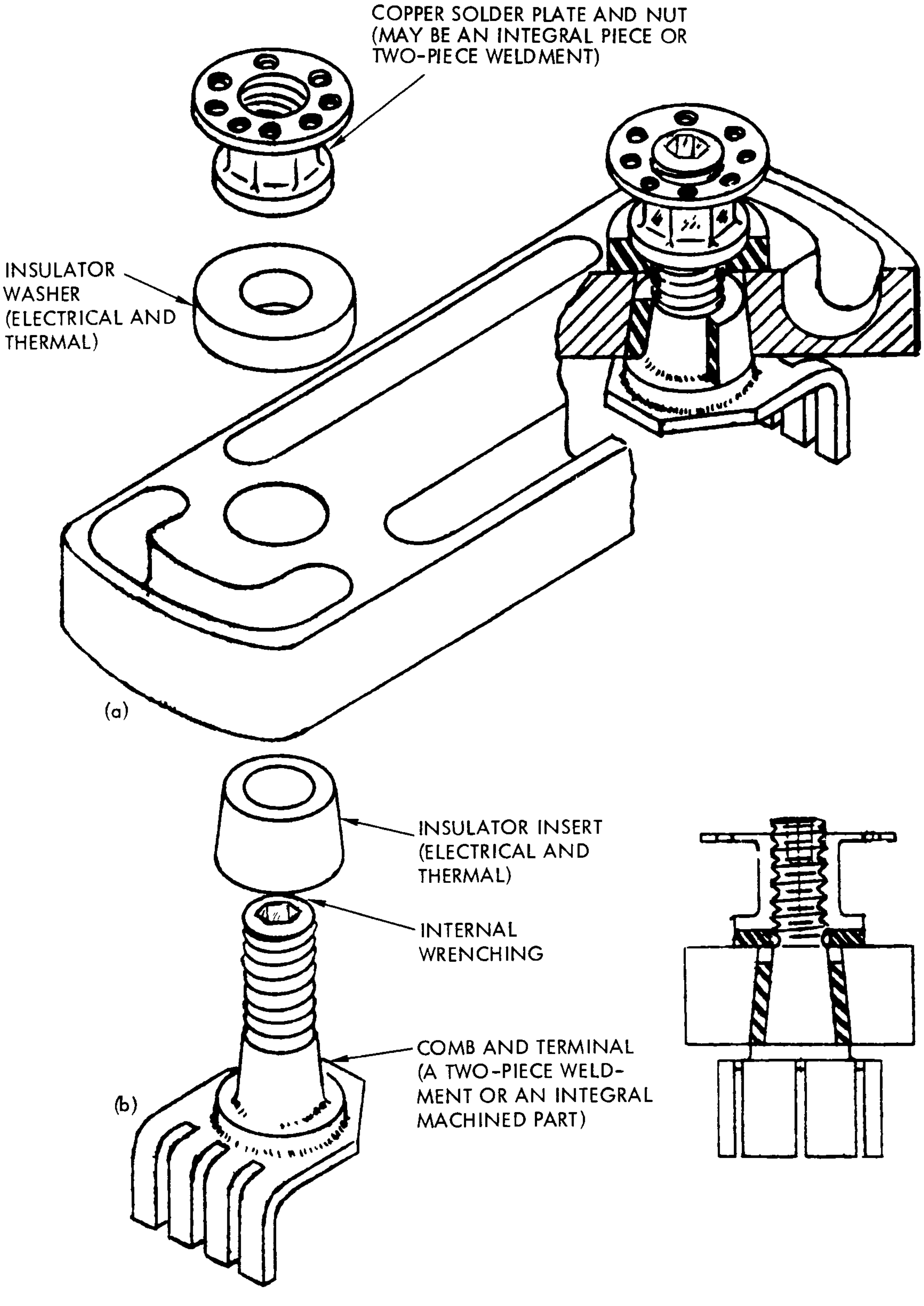

Figure 5-1. Electrically Resistant Cover Design

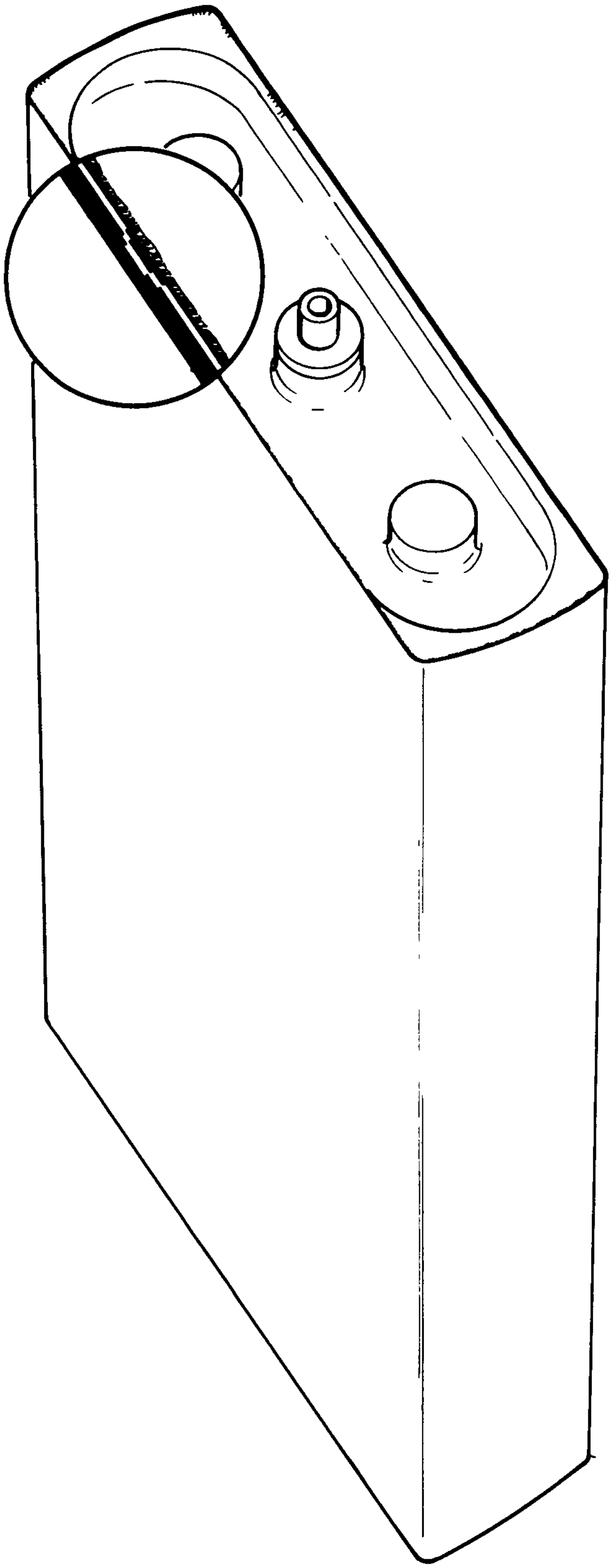

Figure 5-2. Delamination of Case at Cover Interface

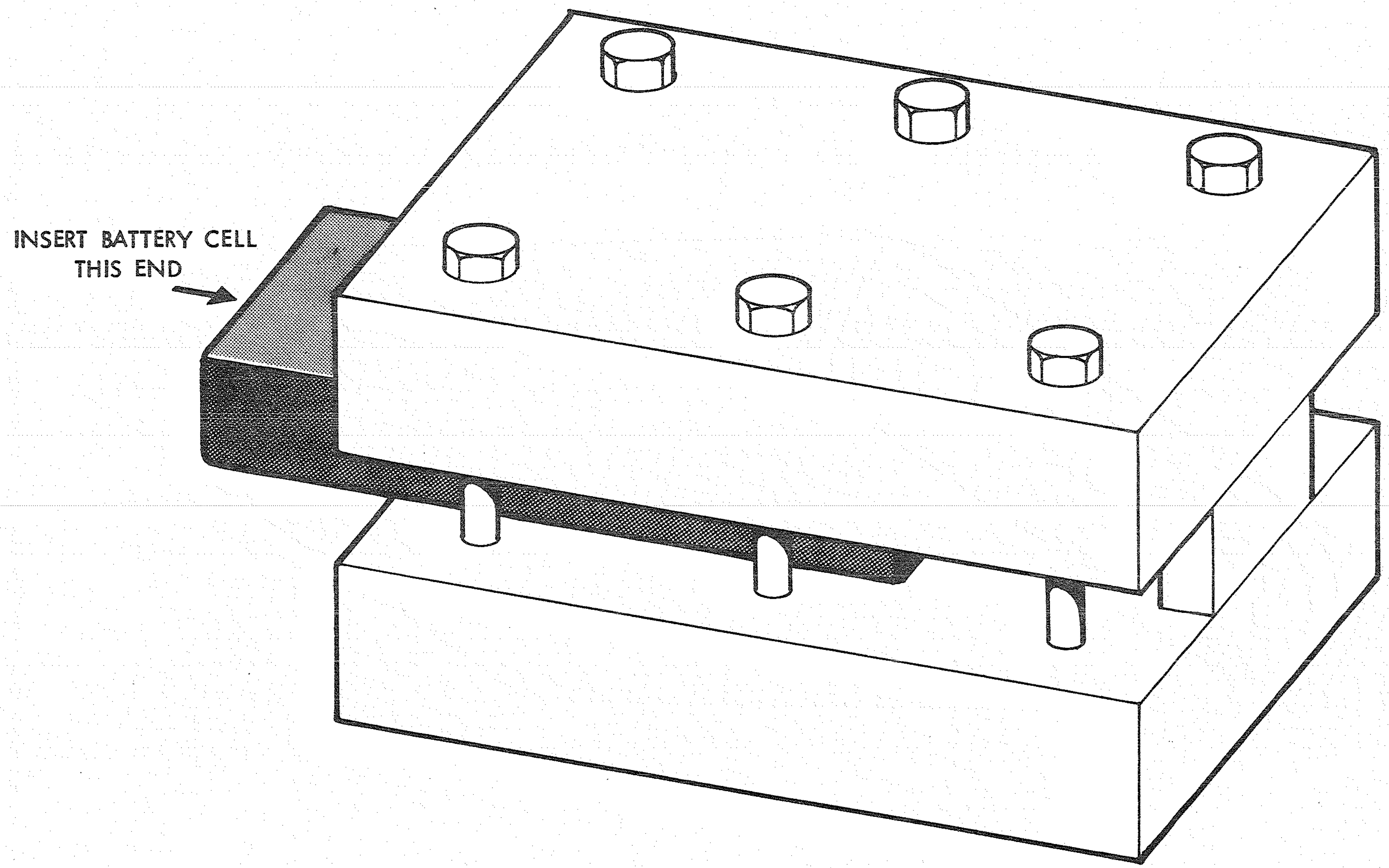

Figure 5-3. Pressure Test Fixture

SECTION VI

DISCUSSION OF RESULTS

Table 6-1 is a tabulation of the various case characteristics monitored throughout the program. The specific gravity and case weight was measured for every case fabricated during the program. The fiber volume and void content were sampled from the initial five cases or each material lot and/or unique manufacturing lots, followed by random selection of cases not exceeding 20 units. The data indicate the following:

(1) The initial processing method utilizing an unreinforced bladder and no interior liner would result in a very low yield (15%) due to rejects for porosity, and erratic burst or proof test values.

(2) Incorporation of secondary operations and rework in addition to restriction of raw material variation and processing parameter tolerance could increase the yield to 60%; however, this would not be cost effective.

(3) Reinforcing the bladder with a metal shim and the lining of Tedlar should increase the yield to the 90% level using standard raw material requirements, and should allow for a high-resin content in the supported broad side of the case.

(4) Control of the raw material resin content and adequate standard quality control monitoring of processing parameters should yield a 95% acceptance of manufactured cases. Closer control of raw material resin content to 43 ±2% would improve yield and case quality.

(5) Monitoring the specific gravity from a tag end of each part and visual inspection should indicate the case with gross defects. Sampling of cases for fiber volume, weight, leakage, and proof pressure should allow the manufacture of cases with confidence to the case specification.

Ten cases that were fabricated by the modified process described herein were selected for burst testing. The results of these burst tests are summarized in Table 6-2. Seven passed the 280 psig design pressure handily, two passed marginally and one case failed.

Twenty subsequent cases have been proof-tested to 100 psig and submitted to environmental exposure. The exposure includes KOH immersion (10 cases) and cyclic testing to 100 psi for 50,000 cycles (10 cases). Upon completion of the environmental exposure, these cases will be burst tested to evaluate the influence of these parameters on projected case life.

Table 6-1. Manufacturing Results

Graphite Epoxy Lot No.	Number of Cases	Specific gravity		Fiber volume		Void Content		Case Weight	Yield
		(0.9 in. side)	(3 in. side)	(0.9 in. side)	(3 in. side)	(0.9 in. side)	(3 in. side)		
08968 Resin Content 43 - 49.4	33	1.55 (0.02)	1.56 (0.02)	58.18 (2.70)	58.84 (4.19)	0.76 (1.75)	0.60 (1.18)	32.86 (1.32)	15
		Failures typified by excessive porosity							
09043 Resin Content 46.9 - 48.7	35		1.57 (0.02)		58.60 (1.86)		0.66 (0.16)	31.87 (1.63)	67
		Failures typified by some porosity and excessive Resin Pudding							
09229 Resin Content 38.8 - 42.7	52		1.57 (0.01)		58.62 (3.42)		0.76 (0.04)	32.19 (2.61)	85
		Failures typified by Corner Porosity							
09336 Resin Content 38 - 45	47		1.57 (0.02)		58.39 (2.25)		0.92 (0.40)	36.53 (1.10)	96
09043 Resin Content	35	1.56 (0.02)	1.53 (0.02)	58.85 (0.70)	58.13 (1.87)	0.91 (0.31)	1.05 (0.72)	39.83 (1.37)	94

Table 6-2. Burst Tests of G/E Cases

Serial number	Burst strength (psi)	Remarks
077	375	Cap/bond failure
078	350	Side-wall fracture
079	350	Side-wall fracture
080	310	Side-wall fracture
081	300	Side-wall fracture
082	350	Side-wall fracture
083	400	Test stopped; no failure
084	400	Test stopped; no failure
085	370	Side-wall fracture
086	230	Side-wall fracture
Average	344	
Average[a]	375	

[a] Less 080, 081, 086

SECTION VII

CONCLUSION AND RECOMMENDATIONS

Once the processing problems encountered during the initial stages of this program were identified and resolved, the remaining 90 battery cases were produced with a minimum of difficulty. The results of the data obtained from these 90 cases indicated that the pressure bladder method for case fabrication is a viable method for manufacturing a reproducible product.

During the course of this development program, four major problem areas were identified and corrected.

(1) Variations in the raw material properties and characteristics. It is recommended that resin content be held to 43-47%.

(2) Imperfections and an irregular internal surface molded with the elastomeric bladder. This condition was improved by incorporating a steel shim into the elastomeric bladder. This created a more uniform distribution of epoxy resin during the cure cycle and, additionally, resulted in a smoothed surface on the interior of the fabricated case. This elimination of localized resin concentrations opened the allowable prepreg resin content tolerance to what is considered industry standard.

(3) Inadequate test fixtures and test procedures. Modification of the test fixture to fully contain the battery case assembly during burst testing and the uniform of pressure improved the previous testing deficiencies.

(4) Localized porosity of the graphite epoxy case wall. Incorporation of a Tedlar film liner eliminated all laminate porosity plus provided another benefit, i.e., increased resistance to concentrated KOH electrolyte.

Recommendations for additional studies that should be considered include:

(1) Development of a Kevlar epoxy battery case. A preliminary evaluation indicates a 35% weight savings over the present G/E battery case.

(2) Manufacturing scale-up for large quantity production. A potential cost reduction of 30-35% could be achieved over present fabrication methods without jeopardizing product uniformity. The three areas of fabrication where cost reductions are attainable are:

(a) Utilization of a high-expansion tool material for case fabrication.

(b) Elimination of all machining on the cover by compression molding of the cover net to print.

(c) Incorporation of machining fixtures for trimming the molded case blank to size.

REFERENCES

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1-2. Bauer, J. L., White, C., and Bogner, S., "Development of Graphite Epoxy Housing for Nickel-Cadmium Cell," Paper presented at the Society for the Advancement of Materials and Process Engineering, Ninth National Technical Conference, Atlanta, Georgia, October 4-6, 1977.

1-3. "Manufacturing Process Specification," Jet Propulsion Laboratory Spec. No. FS511310 Rev. A, October 31, 1977(JPL Internal Document)

2-1. "Material Specification," Jet Propulsion Laboratory, Spec. No. BS506308, May 21, 1975 (JPL Internal Document).

2-2. "Advanced Composites Design Guide," prepared by the Structures Division, Air Force Flight Dynamics Laboratory, Air Force Systems Command, WPAFB, Ohio, September 1976.

2-3. American Society for Testing Materials, Standard Methods of Test for Gas Transmission Rate of Plastic Film and Sheeting, Philadelphia, Pennsylvania, 1978.

5-1. Garcia, A., "Spacers for Graphite Epoxy Cell Testing," IOM 354/AG/067, Jet Propulsion Laboratory, California Institute of Technology, Pasadena, Calif., May 25, 1978 (JPL internal document).

APPENDIX A

MANUFACTURING AND QUALITY CONTROL PLAN FOR FABRICATION OF GRAPHITE FIBER/EPOXY BATTERY CELL CASES

(Prepared by Programmed Composites, Inc., Fullerton, California)

MANUFACTURING AND QUALITY CONTROL PLAN FOR
FABRICATION OF GRAPHITE FIBER/EPOXY
BATTERY CELL CASES

Drawings:	10033546 Rev. A
Specifications:	Graphite filament, preimpregnated, epoxy resin, detail specification BS506308 Rev. A Issue Date 21 May, 1975
	Fabrication of graphite fiber epoxy resin Battery Cell Cases, detail specification for FS511310 Rev. A Issue Date 31 October, 1977
Materials:	Battery Cell Case (P/N 10083546-101) Preimpregnated graphite fiber/epoxy--Ferro Corp. CE 9015/T300 2.5 mil thickness--resin content 45 ±5% 4.0 mil thickness--Tedlar Film 400 XRB160SE--E. I. Dupont Silicone rubber--General Electric Co. RTV 630 Parting agent--Ram Chemical Garan 225 --fluorocarbon spray MS-122 Solvent--methyl ethyl ketone (MEK) Tetra-Etch--W. L. Gore & Assoc.
	Battery Cell Cover (P/N 1008546-102) Molding compound--Ferro Corp CE 9015/T300 1-inch fiber length --U.S. Polymeric EM-7125/T300 1-inch fiber length Parting agent--Ram Chemical Garan 225
	Case Mold Assembly (P/N 77001)
	Cover Mold Assembly (P/N 77002)

Programmed Composites Company 1234 E. Ash Street Fullerton Ca. Sheet 1 of 7

Customer Code	Sales Order	Quantity	Date	Part Name	Part Number
JG686860			11/1/78	Battery Cell Case	10083546-101
Mfg Eng Approval	Q C Approval	Proj Eng Approval	Prod Planner	E.O.'s	Mfg. Order Number Dash

REVISIONS

Date	Originator	Description - Effectivity	ME	QC	PE

Oper No	Code No	OPERATION DESCRIPTION	Oper	Insp
10		Verify and log purchase order numbers for materials.		
		Battery cell case (P/N 10083546-101) fabrication of silicone bladder.		
20		Clean tool (P/N 77001) to remove all foreign materials.		
		Mix 44.0 Grams of RTV 630 as follows:		
		Part A--40 Grams		
		Part B--4 Grams		
		Vacuum deareate the mix.		
		Coat the two main cavity halves of tool (P/N 77001) with a thin layer of RTV mix (050") and place a 3 x 8 1/4 inch 10 Mil steel shim onto the silicone mix. Press into place and cure 30 minutes at 350°F.		
		Remove tool from press and allow to cool to room temperature.		
		Trim silicone rubber approximately 1/8" over size of 3 x 8 1/4 metal shim. Do not release metal/silicone preform from tool.		
30		Assemble the two main cavity halves without center shim.		
		Assemble top spacing shims used for locating of plunger.		
		Mix 220.0 Grams of RTV 630 using the following proportions:		
		Part A--200.0 Grams		
		Part B--20.0 Grams		
		Vacuum deareate the mix.		
40		Charge the mix into the room temperature mold cavity. Insert plunger and clamp. Index to insure plunger is properly centered.		
50		Cure bladder according to the following cycle:		
		1. Cure clamped mold in vertical position for 24 hours minimum at room temperature.		
		2. Place clamped mold, in vertical position, in a room temperature forced air oven (alternate method shall be to remove clamps and place tool into a room temperature platen press. Clamp with press at a load of 3,000 pounds).		

Quantity Accepted	Quantity Rejected	Disposition of Rejections	Inspector	Date

Customer Code	Sales Order	Quantity	Date	Part Name	Part Number
JG686860			11/1/78	Battery Cell Case	10083546-101
Mfg Eng Approval	Q C Approval	Proj Eng Approval	Prod Planner	E O 's	Mfg Order Number Dash

REVISIONS

Date	Originator	Description - Effectivity	ME	QC	PE

Oper No	Code No	OPERATION DESCRIPTION	Oper	Insp
		3. Increase temperature of oven (alternate method press platens) to 300° + 10°F.		
		4. On reaching 300 + 10°F, remove mold, unclamp, and remove bladder on plunger as assembly. Using removal fixture withdraw the plunger from the bladder.		
		5. Postcure the unrestrained bladder in a forced air oven stabilized at 350 ± 10°F for 4 to 6 hours.		
60		Q.C. Verify mix of the RTV 630 and the cure cycle parameters (time and temperature).		
70		Package bladder in clean polyethylene bag and mark with serial number.		
80		Repeat operations 20-70 as required.		
90		Remove preimpregnated graphite fiber/epoxy from 0° storage. Record time and date of removal on storage log. NOTE: DO NOT UNSEAL PACKAGE UNTIL MATERIAL HAS STABILIZED AT AMBIENT TEMPERATURE.		
		vendor data test results		
		. Lot #		
		Prepreg--resin content		
		resin flow		
		volatile content		
		gel time		
		Cured		
		Material-ply thickness		
		flexural strength		
		flexural modulus		
		specific gravity		
		fiber volume		
		void content		

Quantity Accepted	Quantity Rejected	Disposition of Rejections	Inspector	Date

Programmed Composites Company 1234 E. Ash Street Fullerton Ca.

Customer Code	Sales Order	Quantity	Date	Part Name	Part Number
JG686860			11/1/78	Battery Cell Case	10083546-101
Mfg Eng Approval	Q C Approval	Proj Eng Approval	Prod Planner	E.O.'s	Mfg Order Number Dash

REVISIONS

Date	Originator	Description - Effectivity	ME	QC	PE

Oper No	Code No	OPERATION DESCRIPTION	Oper	Insp
		Select a sample at random from the material and test for resin content, resin flow, gel time, and volatile content. Log results into Operation 90.		
110		Fabricate a 4 inch x 4 inch eight-ply laminate (0,90,0,90,90,0,90,0 orientation). Press cure the laminate as follows:		
		1. Place into a platen press stabilized at 380 $\pm$ 10^{o}F and close to contact.		
		2. Dwell 5 minutes.		
		3. Increase pressure to 100 PSI.		
		4. Cure for 60-70 minutes.		
		Remove laminate and test for cured ply thickness, specific gravity, fiber volume, and void content. Log results into Operation 90.		
120		Cut preimpregnated graphite fiber/epoxy ply kits for the Battery Cell Case as follows:		
		4 ea 3 x 19 inch fiber direction 19, ply A		
		4 ea 1 x 21 inch fiber direction 21, ply B		
		4 ea 8 1/2 x 9 * inch fiber direction 8 1/2, ply C		
		* Ply is composed of 3 ea 8 1/2 long by 3 inch wide sections of 3 inch wide prepreg tape.		
		Seal kit in clean polyethylene bag and mark with serial number.		
		Return kit to 0^{o} storage and log date and time on storage log.		
		Reseal unused material and return to 0^{o} storage. Log date and time on storage log.		
130		Q.C. Verify ply dimensions and fiber orientation. Verify out time of material as logged on storage log.		
135		Fab Tedlar bladder as follows:		
		NOTE: TEDLAR BLADDER TO BE HANDLED WITH WHITE GLOVES ONLY.		
		1. Place bladder over aluminum holding fixture and release outer surfaces with MS-122.		
		2. Form precut 9 x 10 inch, 4-Mil Tedlar film over bladder to form liner.		

Quantity Accepted	Quantity Rejected	Disposition of Rejections	Inspector	Date

Customer Code	Sales Order	Quantity	Date	Part Name	Part Number
JG686860			11/1/78	Battery Cell Case	10083546-101

Mfg Eng Approval	Q C. Approval	Proj Eng Approval	Prod Planner	E O's	Mfg Order Number	Dash

REVISIONS

Date	Originator	Description - Effectivity	ME	QC	PE

Oper No	Code No	OPERATION DESCRIPTION	Oper.	Insp
		3. Heat seal Tedlar (300°F).		
		4. Inspect to insure all heat sealed semas are on the .890 inch side wall.		
140		Layup Battery Cell Case as follows: 1. Remove ply kit from 0° storage. (NOTE: DO NOT UNSEAL PACKAGE UNTIL MATERIAL HAS STABILIZED AT AMBIENT TEMPERATURE.) Log kit number and removal time in storage log. 2. Layup part in accordance with the following sequence which is illustrated in Figure 1. Ply 1A, Ply 1B, 2C,3A,4B, 4C, 5C, 6A, 6B, 7C, 8A, 8B. 90° plies will terminate with a 1/2 inch lap joint at the corner and shall be rotated on corner of each additional ply. Gaps in material and at butt joints shall be minimized with a maximum acceptable gap of 0.050 inch. 3. Remove aluminum holding fixture. 4. Preform/bladder may be sealed in a polyethylene bag, marked with serial number, and returned to 0° storage at this stage. Log date and time in storage log.		
150		Q.C. Verify logging of serial numbers and layup sequence.		
160		Clean tool (P/N 10083546-101) to remove all foreign material. Release tool with Garan 225. Assemble preform/bladder into female tool composed of two main cavity halves and spacing shim. Insert bladder toe support assembly. Install face plate and air inlet hose. Load mold assembly into a platen press stabilized at 380 ± 10°F and clamp with 2 tons.		
170		Cure part according to the following schedule: 1. Pressurize bladder to 5 PSIG and maintain for 6 ± 1 minute 2. Increase bladder pressure to 100 PSIG and maintain for 60-90 minutes. Log actual cure parameters on log.		

Quantity Accepted	Quantity Rejected	Disposition of Rejections	Inspector	Date

Programmed Composites Company 1234 E. Ash Street Fullerton Ca.

Customer Code	Sales Order	Quantity	Date	Part Name	Part Number
JG686860			11/1/78	Battery Cell Case	10083546-101

Mfg Eng Approval	Q C Approval	Proj Eng Approval	Prod Planner	E O's	Mfg Order Number	Dash

REVISIONS

Date	Originator	Description - Effectivity	ME	QC	PE

Oper No	Code No	OPERATION DESCRIPTION	Oper	Insp
180		Q. C. Verify cure parameters (pressures, times, and temperatures).		
190		Release air pressure, remove end cap manifold, remove base clamping mandrel, unclamp press and remove tool. Disassemble tool and remove part and bladder as an assembly. Cool assembly to ambient temperature and remove bladder.		
200		Using a 325 grit, 3 inch diameter diamond cutting wheel turning at 4200 rpm, machine Battery Cell Case per Dwg 10083546. Retain and identify cell case and tag end.		
210		Q. C. Verify dimensions of machined Battery Cell Case. Weigh case and record on log.		
220		Assemble case into leak test fixture and pressure to 5 PSIG. Immerse in water bath and observe for 3 minutes minimum. Note location of all leaks.		
230		Q. C. Verify pressure and time of test and logging of leaks.		
240		Test tag end for specific gravity and log results. The first 5 tag ends and tag ends from every 20th part thereafter, will be analyzed as to fiber volume and void content. Log actual test results.		
250		Q. C. Verify specific gravity to conform to 1.53 $\pm$ .05, the fiber volume to 58 $\pm$ 3% and the void content to 3% maximum.		

Quantity Accepted	Quantity Rejected	Disposition of Rejections	Inspector	Date

Programmed Composites Company 1234 E. Ash Street Fullerton Ca. Sheet 6 of 7

Customer Code	Sales Order	Quantity	Date	Part Name	Part Number
JG686860			11/1/78	Battery Cell Cover	1007546-102

Mfg Eng Approval	Q C Approval	Proj Eng Approval	Prod Planner	E O.'s	Mfg Order Number	Dash

REVISIONS

Date	Originator	Description - Effectivity	ME	QC	PE

Oper No	Code No	OPERATION DESCRIPTION	Oper	Insp
		BATTERY CELL COVER (P/N 1008546-102) FABRICATION.		
260		Clean tool (P/N 77002) to remove all foreign material and release with Carnuba wax.		
270		Stabilize a platen press at 325 ± 10°F and adjust clamp pressure to 6 tons. Insert tool and stabilize at 325 ± 10°F.		
280		Weigh out 12.7 Grams of molding compound (U.S. Polymeric EM-7125) and charge heated tool.		
290		Cure Battery Cell Cover blank as follows: 1. Close press to contact pressure and dwell for 45-60 seconds. 2. Release pressure and open press 1/4 inch. 3. Close press and apply 6 tons. 4. Maintain pressure and temperature for 45 minutes. 5. Release pressure and remove tool. 6. Disassemble tool and remove part by removing bottom plate, lock plunger retaining screw, eject part and ejector pins through bottom of cavity, unlock plunger retaining screw, and eject plunger through bottom of cavity. Log actual process parameters.		
300		Q. C. Verify molding parameters. (charge weight, times, temperature, and pressure).		
310		Select finished Battery Cell Case and machine cover to allow a bond line on assembly of .004 inch. Package case and cover together. Weigh cover and record weight on process log.		
320		Q. C. Verify bond line dimension and cover weight.		

Quantity Accepted	Quantity Rejected	Disposition of Rejections	Inspector	Date

Programmed Composites Company 1234 E. Ash Street Fullerton Ca. Sheet 7 of 7

Customer Code	Sales Order	Quantity	Date	Part Name	Part Number
JG686860			11/1/78	Battery Cell Cover	1008546-102

Mfg Eng Approval	Q C. Approval	Proj Eng Approval	Prod Planner	E O 's	Mfg Order Number	Dash

REVISIONS

Date	Originator	Description - Effectivity	ME	QC	PE

Oper No	Code No	OPERATION DESCRIPTION	Oper	Insp
		MARKING, CONDITIONING AND PACKAGING.		
330		Mark Battery Cell Case in 1/8 inch high white letters with part and serial number. Mark cover with case serial number.		
340		Clean cases with MEK and lightly sand case and cover bond surfaces with 600 grit paper.		
350		Q. C. Verify cleaning and preparation of bond surfaces.		
360		Condition Battery Cell Case and Battery Cell Cover in 250 $\pm$ 10^{o}F stabilized forced air oven for 4 hours minimum. Cool to ambient temperature and immediately seal in moisture proof polyethylene bag.		
370		Q. C. Verify conditioning and packaging (time and temperature).		

Quantity Accepted	Quantity Rejected	Disposition of Rejections	Inspector	Date

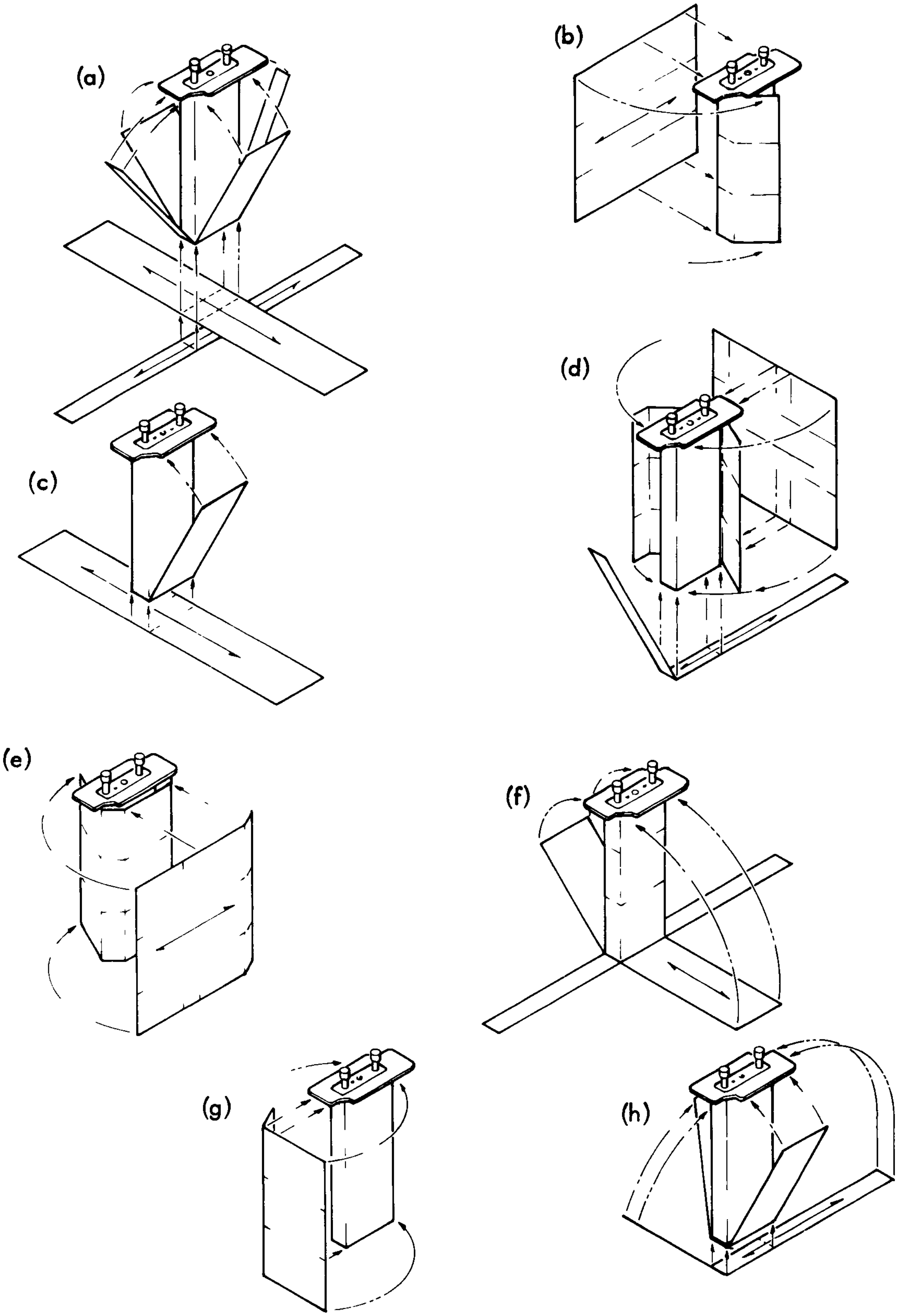

Figure A-1. Manufacturing Sequence

APPENDIX B

DETAILED DESIGN FOR ELECTRICALLY INSULATING COVER

JET PROPULSION LABORATORY INTEROFFICE MEMO

April 11, 1978

TO: J. Bauer

FROM: R. H. Dawe/A. Delgadillo

SUBJECT: Design Idea for Graphite Epoxy Battery Case Terminal

Pursuant with your request, sketches of a best guess terminal design to support your vendor are enclosed. No development was done to establish key parameters necessary for implementation of the design.

The design was to provide for 20 amp continuous electrical current with 200 amp pulses, solder connections for external wire attachments, interface to battery cell same as old header (plate area and head clearance), and material selection per your specification.

The primary areas of concern in the design were to maintain a seal through the various temperature and pressure environments, external lead attachment and removal without degrading the seal, and assuring adequate electrical performance.

The soldered lead attachment and removal was designed to provide sufficient thermal gradient with normal soldering techniques to prevent losing the seal. However, the material characteristics to check this analytically were not furnished. The teflon seal was designed to completely fill the void in the cover and constrained to prevent cold flow after the initial thermal conditioning.

Cell terminal attachment and alignment was specified as not being a design consideration for this concept.

RHD/AJD:jl

Enclosure: SK AD041078

cc: A. Franzon
W. Read
C. Savage
J. Schmuecker

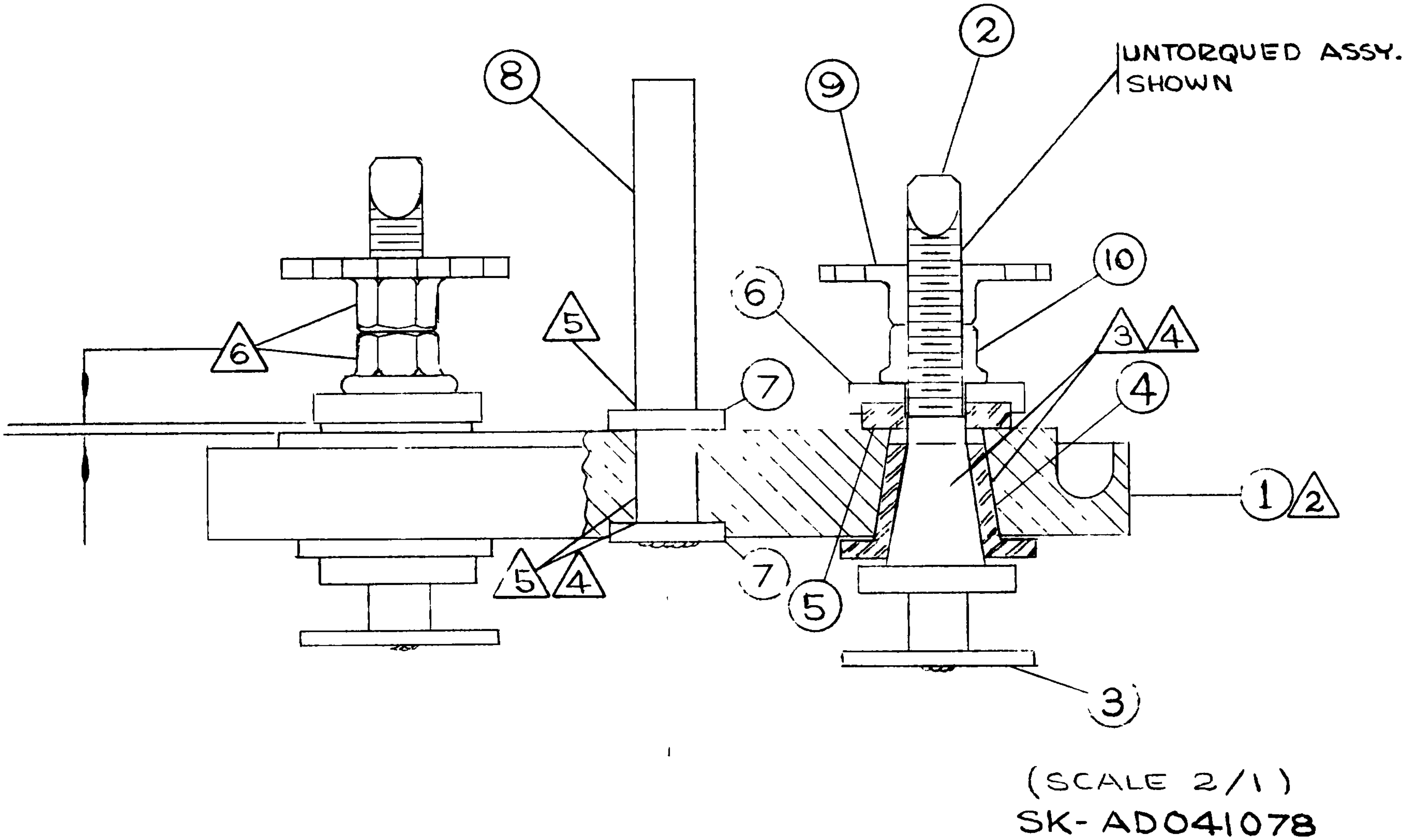

Figure B-1. Terminal Design for G/E Cover Assembly

QTY. REQD	ITEM NO.	PART OR IDENT NO.	NOMENCLATURE OR DESCRIPTION	SPEC'S	MATERIAL OR NOTE
1	1	-101	COVER	BS506308	2
2	2	-102	TERMINAL		NICKEL 270 6 8
2	3	-103	COMB PLATE		NICKEL 270
2	4	-104	INSERT	L-P-389	FEP TEFLON
2	5	-105	ISOLATION WASHER	E540-(TBD)	E PR. RUBBER 6
2	6	-106	CUP WASHER	BS506334	321 STAINLESS
2	7	-107	WASHER	BS506334	321 STAINLESS 5
1	8	-108	VENT TUBE	BS506334	321 S S. TUBE 5
2	9	-109	SOLDER PLATE NUT		NICKEL 270 7 9
2	10	MS35649-204	NUT, HEX	10-32	6
A/R	11	Sn 63/PB37	BAR SOLDER	QQ-S-571	7

Figure B-1. (Contd)

NOTES: UNLESS OTHERWISE SPECIFIED

1. FABRICATE PER JPL SPEC FS51130.

2. FABRICATE [illegible] FROM NARMCO 5208/T-300 CHOPPED RANDOM FIBER, MOLDING COMPOUND.

3. RANDOMLY ROUGH INTERFACE SURFACES WITH 360 GRIT EMERY PAPER

4. APPLY ADHESIVE 3M EC 2216 CLEAN INTERFACE SURFACES PRIOR TO INSTALLATION AND TORQUING.

5. PRESS FIT ASSEMBLY TOGETHER WITH A .0005" INTERRUPT FIT WHICH WILL BE DEPENDENT ON THE SELECTED TUBE O.D.

6. TORQUE EACH NUT INDEPENDENTLY WHILE HOLDING THE FLATS OF THE TERMINALS AS FOLLOWS:
   1. RUN NUT (10) DOWN TERMINAL UNTILL IT IS FINGER TIGHT AGAINST CUPED WASHER (6)
   2. RECORD THE GAP BETWEEN THE WASHER (6) AND COVER (1)
   3. TORQUE THE NUT UNTILL THERE IS A .024 IN COMPRESSION OF THE RUBBER WASHER AND A 55 IN LBS VALUE IS REACHED. (SELECT SHORE OF RUBBER AS REQUIRED).
   4. TEMP. CYCLE TORQUED ASSEMBLY FROM –___°F TO +___°F, RETURN TO AMBIENT AND CHECK FOR LOSS OF TORQUE. REPEAT FOR A MIN. OF 3 CYCLES AFTER THE TORQUE HAS STABILIZED.

7. SOLDER COAT USING TYPE A FLUX, MIL-F-14256 AND BAR SOLDER SN 63/PB37, QQ-S-571, AND FUSE.

8. LIQUID HONE SURFACE EVENLY FOR A 125 RMS FINISH

9. HOLE DIA IS SIZED FOR A 12 GAGE WIRE AS SHOWN (HOLE DIA CAN BE ADJUSTED TO OTHER WIRE SIZES)

10. DIMENSIONS ARE IN INCHES, DO NOT SCALE THE SKETCH

11. SCALE IS CALLED OUT ON EACH SKETCH

12. MACHINE FINISH 32 TYP

Figure B-1. (Contd)

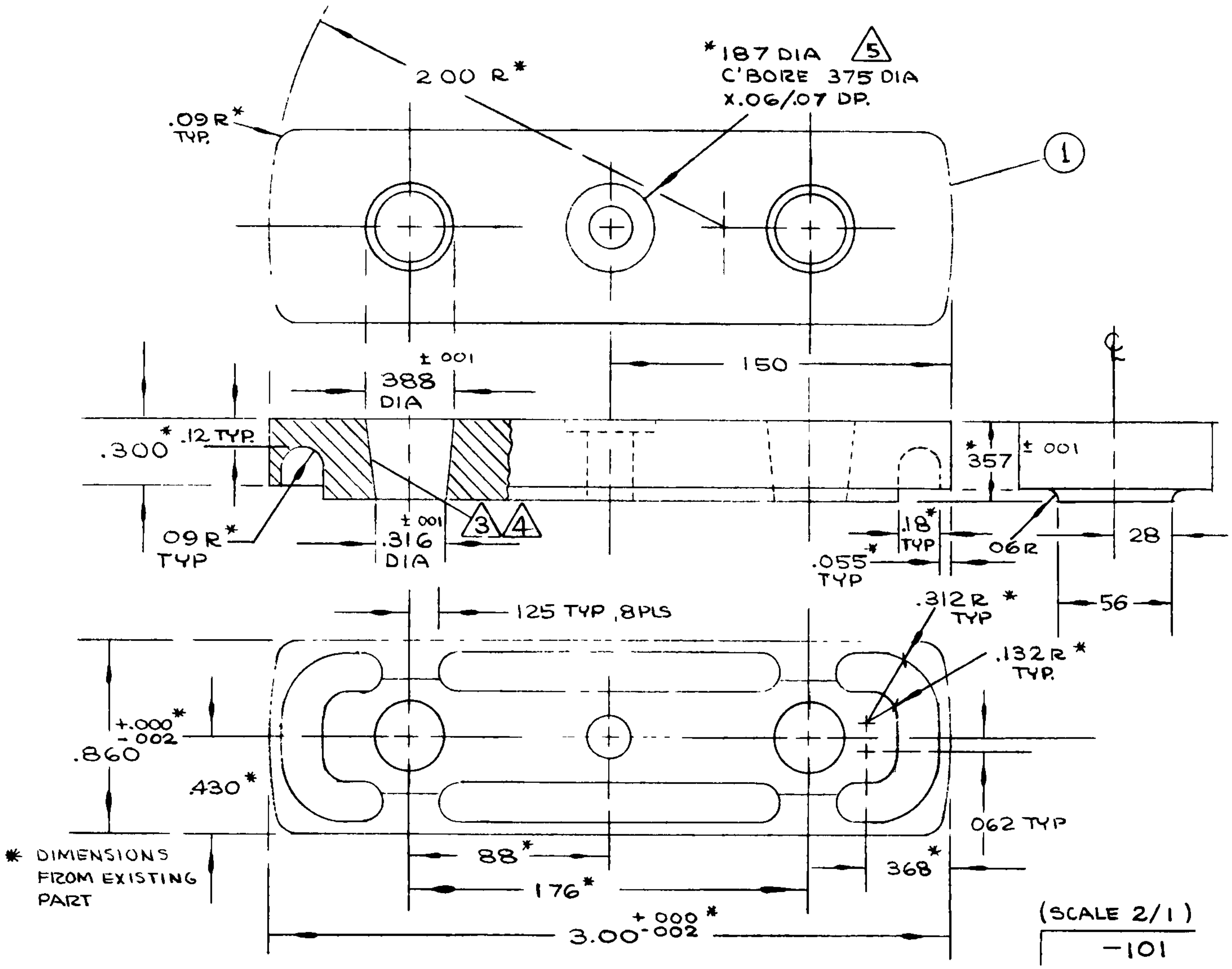

Figure B-2. Detail -101

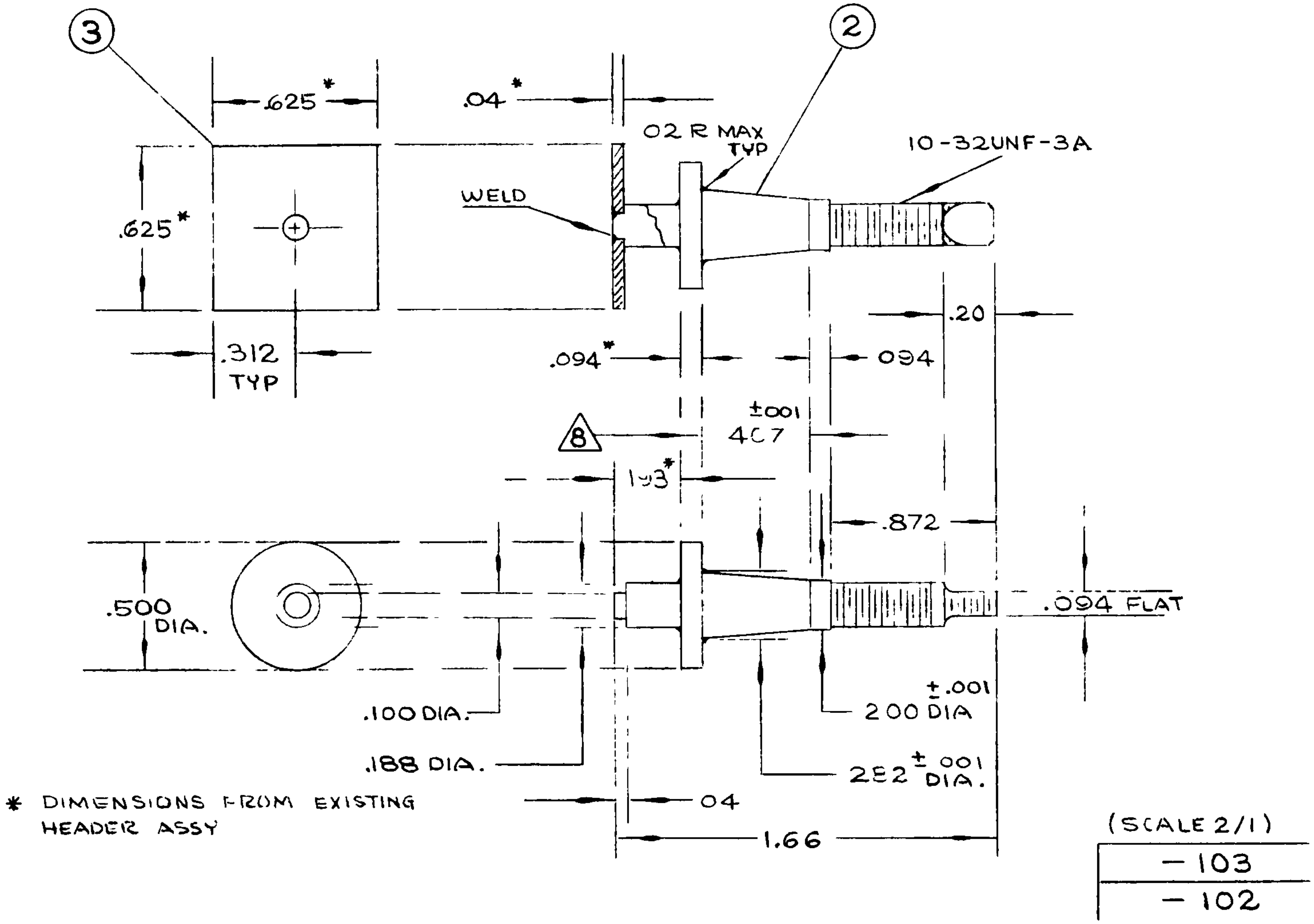

Figure B-3. Details -102 and -103

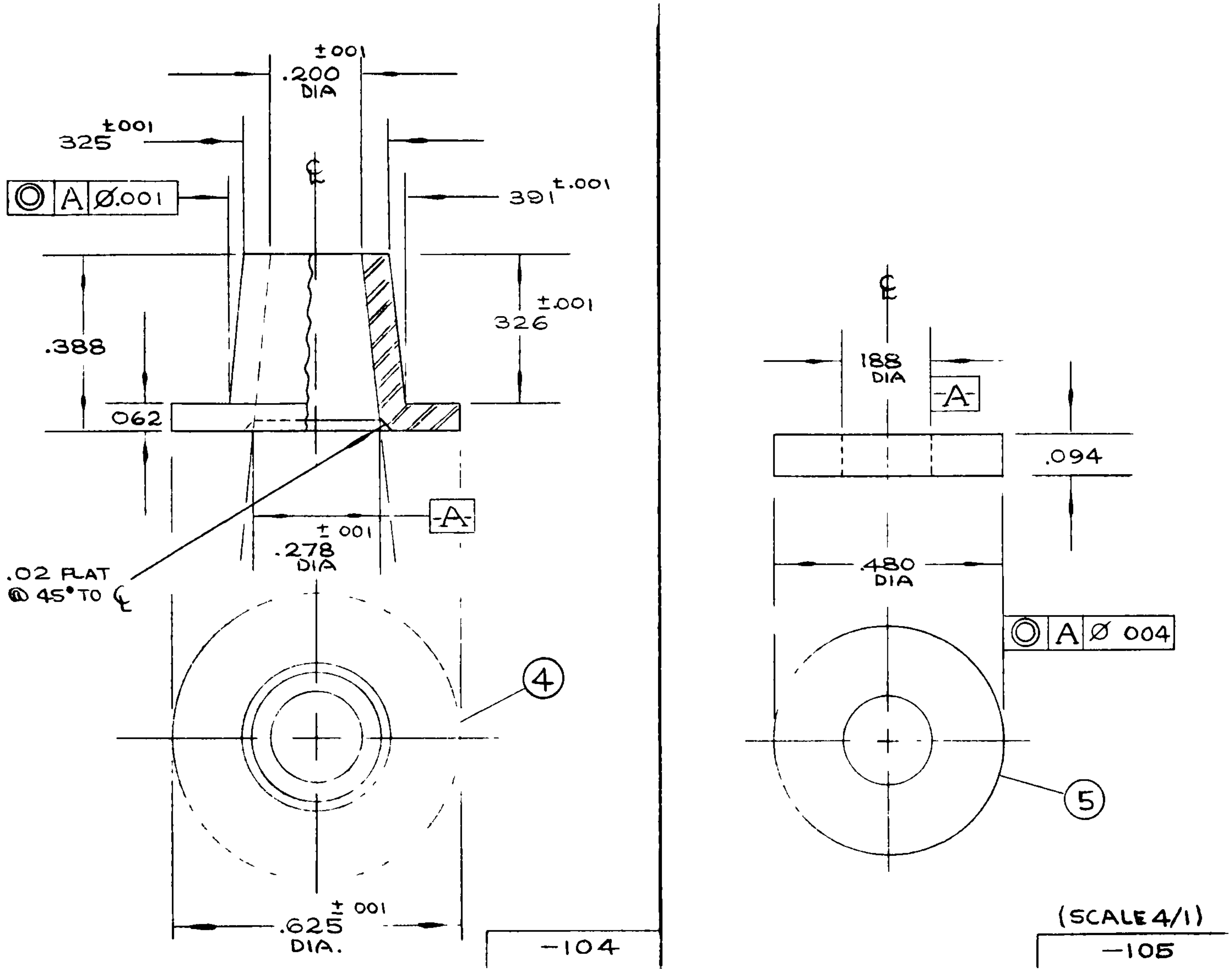

Figure B-4. Detail -105

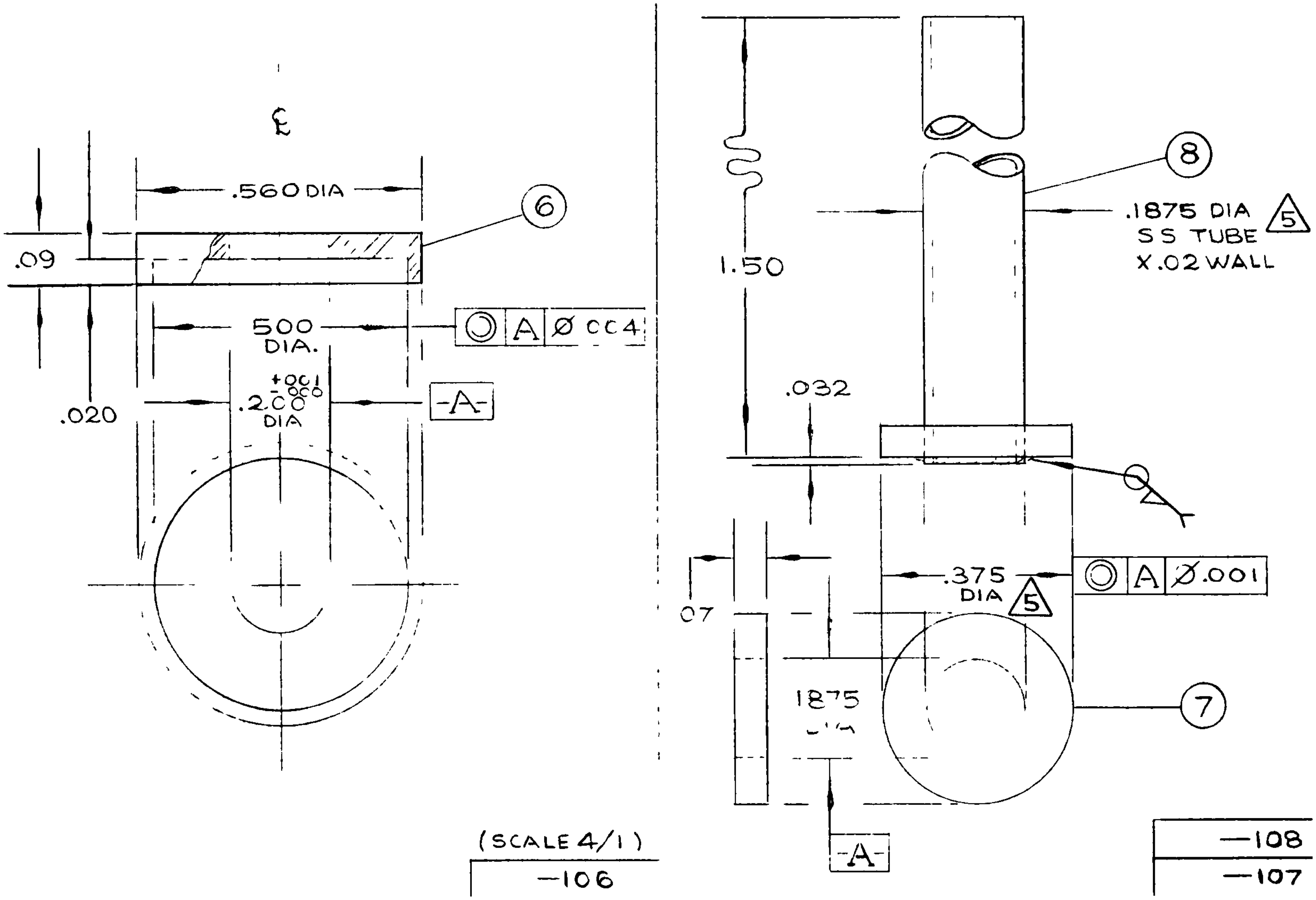

Figure B-5. Detail -107 and -108

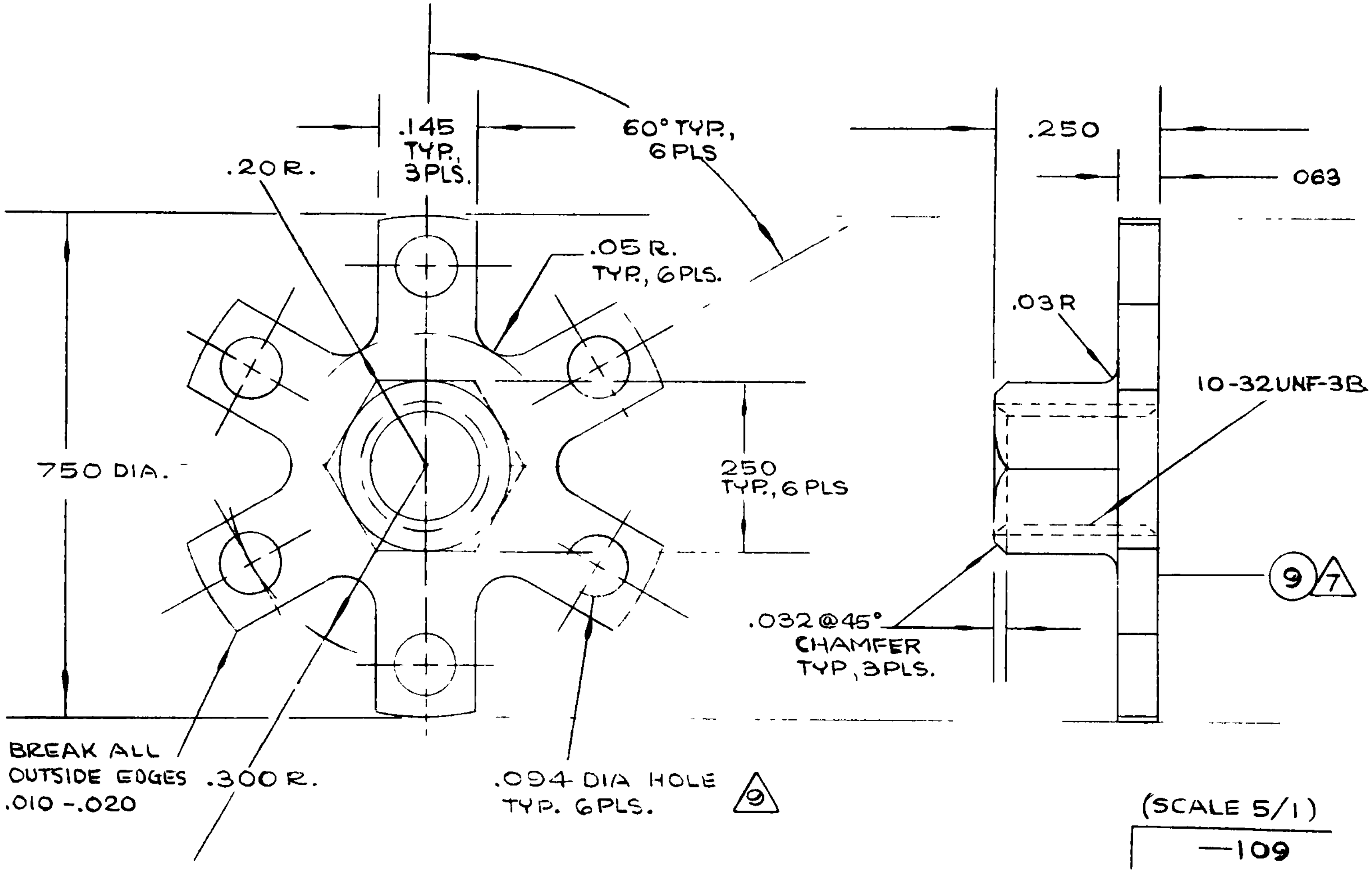

Figure B-6. Detail -109

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